MCR-72-130
Contract NAS8-25974

Final
Report

July 1972

Development of Advanced Material Composites for Use as Internal Insulation for LH_2 Tanks (Gas Layer Concept)

Prepared for
George C. Marshall
Space Flight Center,
Marshall Space
Flight Center,
Alabama 35812

(NASA-CR-124222) DEVELOPMENT OF ADVANCED MATERIAL COMPOSITES FOR USE AS INTERNAL INSULATION FOR LH2 TANKS (GAS LAYER CONCEPT) Final Report (Martin Marietta Corp.) 131 p HC $8.75 CSCL 11D G3/18

N73-22528

Unclas
16055

MARTIN MARIETTA

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.

4. Title and Subtitle

DEVELOPMENT OF ADVANCED MATERIAL COMPOSITES FOR USE AS INTERNAL INSULATION FOR LH_2 TANKS (GAS LAYER CONCEPT)

5. Report Date
July 1972

6. Performing Organization Code

7. Author(s)
J. P. Gille

8. Performing Organization Report No.
MCR-72-130

9. Performing Organization Name and Address
Martin Marietta Corporation
Denver Division
Denver, Colorado 80201

10. Work Unit No.

11. Contract or Grant No.
NAS8-25974

12. Sponsoring Agency Name and Address
National Aeronautics and Space Administration
Washington, D.C. 20546

13. Type of Report and Period Covered
Contractor Report

14. Sponsoring Agency Code

15. Supplementary Notes Prepared for George C. Marshall Space Flight Center, Marshall Space Flight Center, Alabama 35812
Technical Contract Monitor, Dr. James M. Stuckey, MSFC Astronautics Laboratory

16. Abstract

This report describes a program that was conducted by Martin Marietta Corporation to develop an internal insulation system for potential application to the liquid hydrogen tanks of a reusable booster, where the tanks would be subjected to repeated high temperatures. The design of the internal insulation is based on a unique gas-layer concept, in which capillary or surface tension effects are used to maintain a stable gas layer, within a cellular core structure, between the tank wall and the contained liquid hydrogen.

The specific objectives of this program were to select materials for insulation systems that would be compatible with wall temperatures of 350°F and 650°F during reentry into the earth's atmosphere, and to fabricate and test insulation systems under conditions simulating the operating environment.

A materials test program was conducted to evaluate the properties of candidate materials at elevated temperatures and at the temperature of liquid hydrogen, and to determine the compatibility of the materials with a hydrogen atmosphere at the appropriate elevated temperature. The materials that were finally selected included Kapton polyimide films, silicone adhesives, fiberglass batting, and in the case of the 350°F system, Teflon film.

Small insulation system specimens were fabricated using the selected materials, as well as earlier candidate materials. These specimens were subjected to thermal shock tests and combined pressure and temperature cycle tests. The system specimens fabricated with the selected materials, and using the design and fabrication concepts that were developed in this program, demonstrated the capability of the insulation to withstand the repeated high-temperature exposures. The thermal conductivity of the insulation was measured using liquid hydrogen as the test fluid, and was found to be near that for hydrogen gas, as expected, for the temperature range of interest.

17. Key Words (Suggested by Author(s))
Cryogenic insulation
Internal cryogenic insulation
Capillary insulation
Gas-layer insulation
Liquid hydrogen tank insulation

18. Distribution Statement

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price
Unclassified	Unclassified	119	

FOREWORD

This report was prepared by Martin Marietta Corporation under Contract NAS8-25974, *Development of Advanced Material Composites for Use as Internal Insulation for LH_2 Tanks*, for the George C. Marshall Space Flight Center, National Aeronautics and Space Administration. The NASA contract monitor was Dr. James M. Stuckey of the MSFC Astronautics Laboratory.

CONTENTS

Figure

Table

SUMMARY

The objective of this program has been to develop an internal insulation system applicable to hydrogen tanks for reusable space vehicles. The insulation concept that was investigated is based on using surface tension or capillary forces to position a layer of hydrogen vapor in a cellular structure between the liquid hydrogen and the tank wall.

For a resuable vehicle, the insulation must be capable of surviving repeated temperature excursions near the maximum operating temperature of the tank wall. Insulation systems used with tanks of aluminum and high-performance nickel or titanium alloys were evaluated at 350°F and 650°F.

A materials screening program was conducted to identify promising candidates for fabricating the insulation. These candidates were then tested and evaluated. Selected material systems were used to fabricate test specimens and these specimens were tested to measure their performance and ability to survive the simulated vehicle environment. System designs, fabrication methods, and tooling concepts were developed.

The systems that were finally selected used silicone adhesives, Kapton film, and fiberglass batting and--for the case of the 350°F system--Teflon film. In tests with small specimens, the insulating capability of the system was measured to be approximately as expected, and its ability to withstand the required environments was demonstrated.

I. INTRODUCTION

The pioneering phases of the United States' space exploration program have been accomplished with remarkable success. But now, the advanced and continuing phases of space research and exploration present new problems and challenges in designing spacecraft and booster systems. Current objectives of the National Aeronautics and Space Administration include developing reusable vehicles and systems that will provide increased capability for manned space research and exploration at moderate cost.

A major requirement of reusable vehicles is that they be able to withstand high temperatures during atmospheric reentry with only a minimum of damage that would necessitate extensive repair, replacement, or refurbishment. For vehicles with integral boost propellant tanks for liquid hydrogen, these tanks will occupy a large portion of the total volume of the vehicle. Therefore, providing external thermal insulation to protect these tanks from the reentry heating environment will be a major problem because of the large surface areas involved.

The objective of the program described in this report was to develop an internal insulation system based on a unique gas layer concept, which minimizes the need for external thermal protection of integral liquid hydrogen boost tanks for reusable space vehicles. The minimization of thermal protection is accomplished by designing the insulation with a materials system that will permit the tank walls to operate at the maximum temperature at which the metal used in constructing the tank retains useful structural properties. For aluminum alloy tank materials, the desired operating temperature for the insulation was 350°F. For candidate materials with a higher temperature capability, such as titanium or high-performance nickel alloys, the design objective was an insulation that would withstand repeated operating cycles to 650°F.

The insulation systems developed for this application are based on the internal capillary insulation concept illustrated in Fig. I-1. In this concept, the insulation acts to position a layer of gas between the liquid cryogen and the tank wall.

The insulation consists of a cellular core material covered with a face sheet and bonded to the inside surface of the tank. The cells each contain a filler material to limit heat transfer by convection and radiation. The face sheet is perforated with one capillary opening per cell, which permits the movement of gas or liquid to equalize pressure differentials.

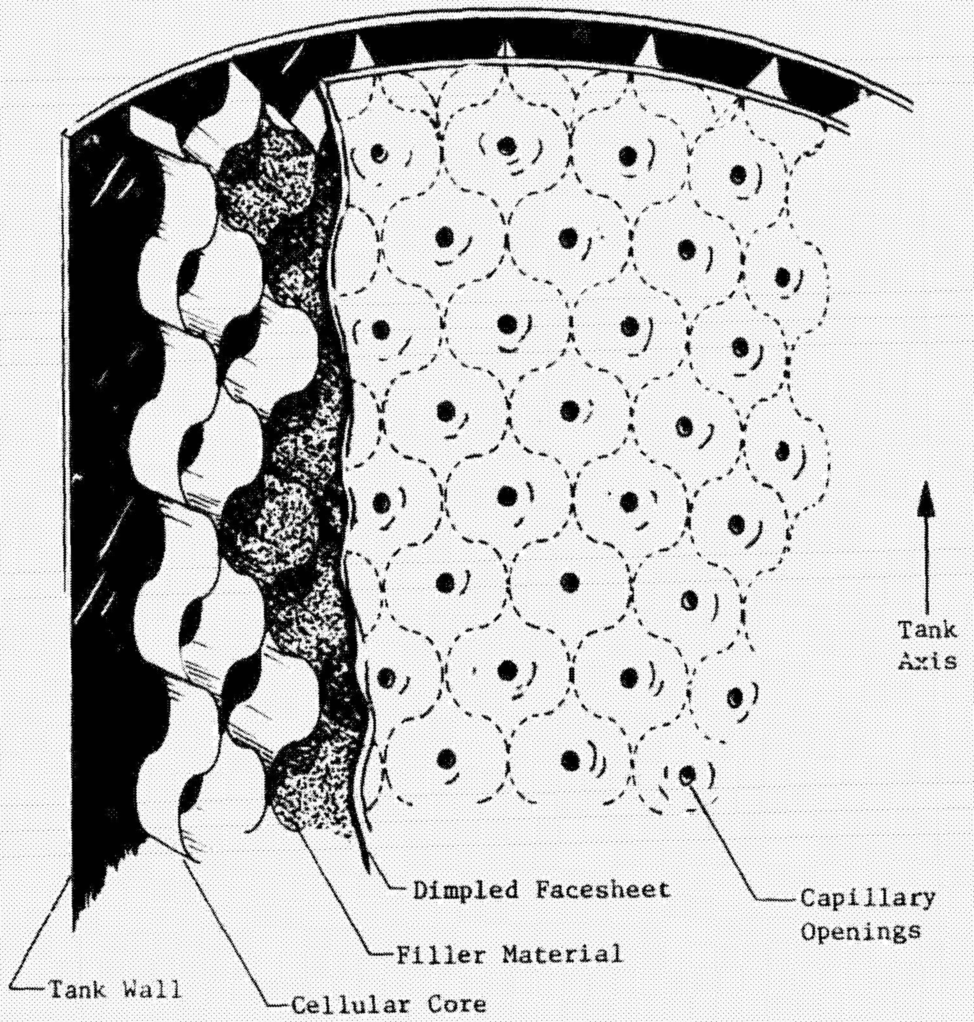


Fig. I-1 Capillary Internal Insulation Concept

A small amount of liquid initially enters the cell, vaporizes, and increases the pressure in the cell until it equals the pressure of the liquid. Capillary forces then act to form a stable liquid/gas interface at the opening, preventing additional liquid from entering the cell. Since the pressure is equalized at this interface, the insulation does not support the weight of the liquid or gas pressure loads.

Because there is no significant pressure difference between the interior of the insulation cell and the adjacent liquid (or gas), structural requirements are low and high strength is not a primary consideration in selecting candidate materials. It has, therefore, been possible to design insulation systems based on this concept using materials with high temperature capabilities. The internal capillary insulation concept and design considerations are discussed in Chapter III.

This insulation system is capable of withstanding severe thermal shock, and the parts of the insulation exposed to the liquid have a very low thermal mass. Consequently, this insulation makes it possible to fill tanks with liquid hydrogen without a prior cooldown. Furthermore, boiloff losses during filling are almost negligible when compared to losses incurred in filling externally insulated tanks.

The insulation is accessible for inspection and repair from the inside and is not subject to damage from external handling and exposure. When cold, the insulation is not exposed to air, and cannot be degraded by condensation of moisture.

This program consisted of three major parts. In the first part, materials were investigated, tested, and selected for use in insulation systems to meet the 350°F and the 650°F operating requirements. Second, designs and methods were developed for fabricating and installing the insulation. Finally, insulation system specimens were tested to evaluate their thermal performance characteristics and to establish their capability to withstand thermal shock and the required pressure and temperature cycling. The work performed to accomplish the above tasks is described in Chapters II, III, and IV.

Two insulation systems were developed and successfully tested at the 350°F and 650°F operating conditions. Performance of the insulation was characterized by measuring its thermal conductivity using liquid hydrogen as the test cryogen. The thermal conductivity was found to be near the predicted value, and completely adequate for the liquid hydrogen boost tank application.

Additional work toward developing the capillary internal insulation concept for other applications has been conducted on the following programs.

1) Contract NAS8-21330, *Development of Advanced Materials for Integrated Tank Insulation System for the Long-Term Storage of Cryogens in Space*;

2) Contract NAS3-12425, *Insulation Systems for Liquid Methane Fuel Tanks for Supersonic Cruise Aircraft*;

3) Martin Marietta Corporation internal venture program to develop an insulation system for storing and transporting liquefied natural gas.

At the present time, the design and fabrication techniques developed under this program are being used and extended under Contract NAS3-14384, *Internal Insulation System Development*. In this program, a 6-ft-diameter by approximately 7-ft-long aluminum tank is being insulated with the 350°F insulation system described in this report. A number of photographs showing tooling and insulation panels that have been fabricated during the 6-ft tank program have been included in this report, and are identified by the above contract number. These examples illustrate the application of the design and fabrication concepts developed under this program (see Chapter III) to the insulation of an actual tank.

II. EVALUATION AND SELECTION OF MATERIALS

A major objective of this program was to select materials systems for use in two potential applications of the capillary internal insulation system. These applications centered around the concept of minimizing external thermal protection for reusable liquid hydrogen tanks for the Space Shuttle.

In the first case, aluminum alloy tanks would be used. The insulation would be designed to permit the tank walls to reach a temperature up to 350°F, which would be the maximum useful temperature for the aluminum. Ideally, the insulation system would be able to withstand repeated exposure to this temperature without being replaced or refurbished.

For the second case, the tank walls would be made of a titanium alloy or Inconel 718. In this case, the goal was to develop an insulation system that would be capable of operation at 650°F.

During this program, we evaluated several candidate materials for various components of the insulation and selected materials systems that meet these temperature requirements.

Our proposed capillary internal insulation system is constructed of the following components:

1) Core ribbon;

2) Facesheet;

3) Core node bond adhesive;

4) Core-to-facesheet adhesive;

5) Core-to-tank wall adhesive;

6) Convection-inhibiting filler meterial.

The following sections describe our program for screening, evaluating, and selecting the materials for the insulation system.

A. MATERIALS SCREENING TEST PROGRAM

In the initial materials screening program, we identified several candidate materials for the 350°F insulation system and the 650°F insulation system and determined their basic properties and hydrogen compatibility. Candidate materials for the high-temperature system had previously been identified under NASA Contract NAS3-12425, *Insulation Systems for Liquid Methane Fuel Tanks for Supersonic Cruise Aircraft*. These materials were further evaluated in this program. The following subsections describe the test methods that were used.

1. Thermal Stability

A differential scanning calorimeter (DSC) analysis in hydrogen gas was performed on some candidate materials. Temperatures ranged from about 70°F to approximately 10-20°F above the melting point of the material, or, in some cases as high as 930°F, which is the upper limit of the machine. Each sample was heated at a rate of 5°C per minute. This analysis was used to determine whether any reactions occurred within the elevated temperature range of interest.

Figure II-1 shows the equipment used for the DSC tests. Figure II-2 is a magnified picture of an open and sealed sample container.

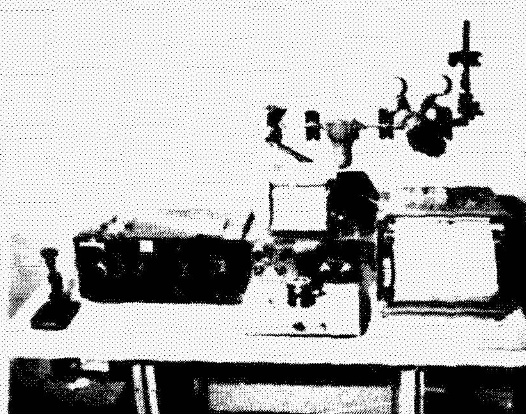


Fig. II-1 Differential Scanning Calorimeter Equipment

Fig. II-2 Differential Scanning Calorimeter Specimens - Open (Left), Sealed (Right)

2. Hydrogen Compatibility

Hydrogen thermal cycle tests were performed by placing the candidate materials in a stainless steel retort, pressurizing them with hydrogen gas to approximately 5 to 10 psig at room temperature, and alternately heating the materials to 350°F or 650°F (depending on the application for which the material was being considered) in a controlled salt bath and cooling them in ambient air.

The specimens were removed from the fixture at the end of 200 cycles (or as otherwise noted), and evaluated by visual inspection, IR spectrography, and measurements of their weight change, dimensional change, and tensile and/or shear properties at room temperature. Figure II-3 shows the hydrogen thermal cycle test fixture. Figures II-4 and II-5 are typical temperature cycles for the 350°F and 650°F insulation materials, respectively.

3. Infrared Spectroscopy

An infrared double-beam spectrographic analysis was performed on transparent core and facesheet materials in the 2 to 15-micron range, using the transmission method. Tests were conducted both before and after hydrogen thermal cycle tests. A comparison of the two spectra was used to measure any chemical reaction during the hydrogen soak test.

4. Tensile Properties

Tensile strength, modulus, and elongation were measured according to Federal Test Standard 406, using Method 1011 for the core material and Method 1013 for the facesheet materials. Tensile properties for the selected candidate materials were measured at room temperature, -423°F, and 350°F or 650°F, depending on the application.

Figure II-6 shows the equipment used for tensile and shear tests at the higher temperatures. Figure II-7 shows the tensile and shear test equipment for -423°F. Figure II-8 shows typical tensile test specimens before and after the test.

5. Shear Properties

Adhesive lap shear tests were conducted according to Federal Test Standard 175. Lap shear specimens were fabricated with a ½-in. overlap. For the 350°F system, a piece of Mylar or Kapton was bonded between the aluminum tabs. For the 650°F system, a piece of Kapton was bonded between the adhesive and titanium or Inconel tabs, as shown in Fig. II-9. Specimens were tested (1) at room temperature, (2) at -423°F, (3) at room temperature after the 200-hr hydrogen compatibility cycle test, and (4) at either 350°F or 650°F, depending on the application. Figure II-10 shows a typical lap shear test specimen before and after the test.

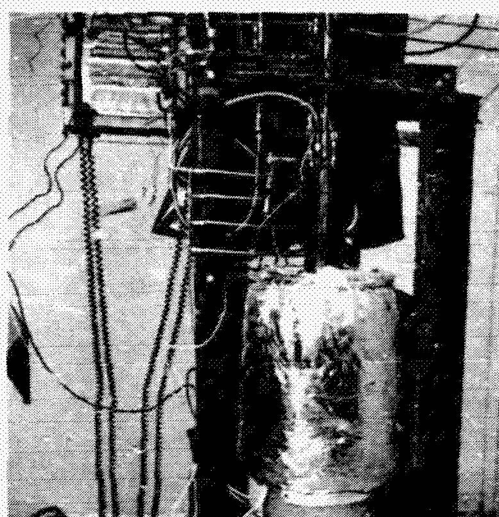


Fig. II-3 200-hr Hydrogen Thermal Cycle Fixture

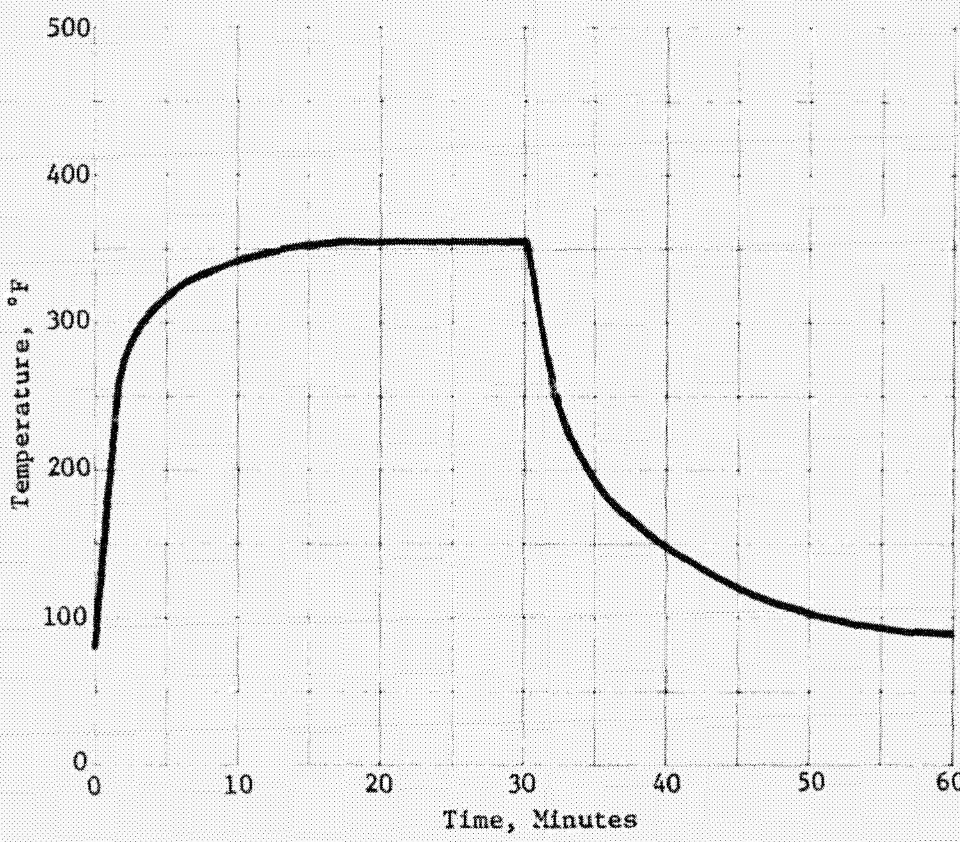


Fig. II-4 Temperature vs Time Profile of 350°F Insulation Materials During Cycle in Hydrogen Soak Test

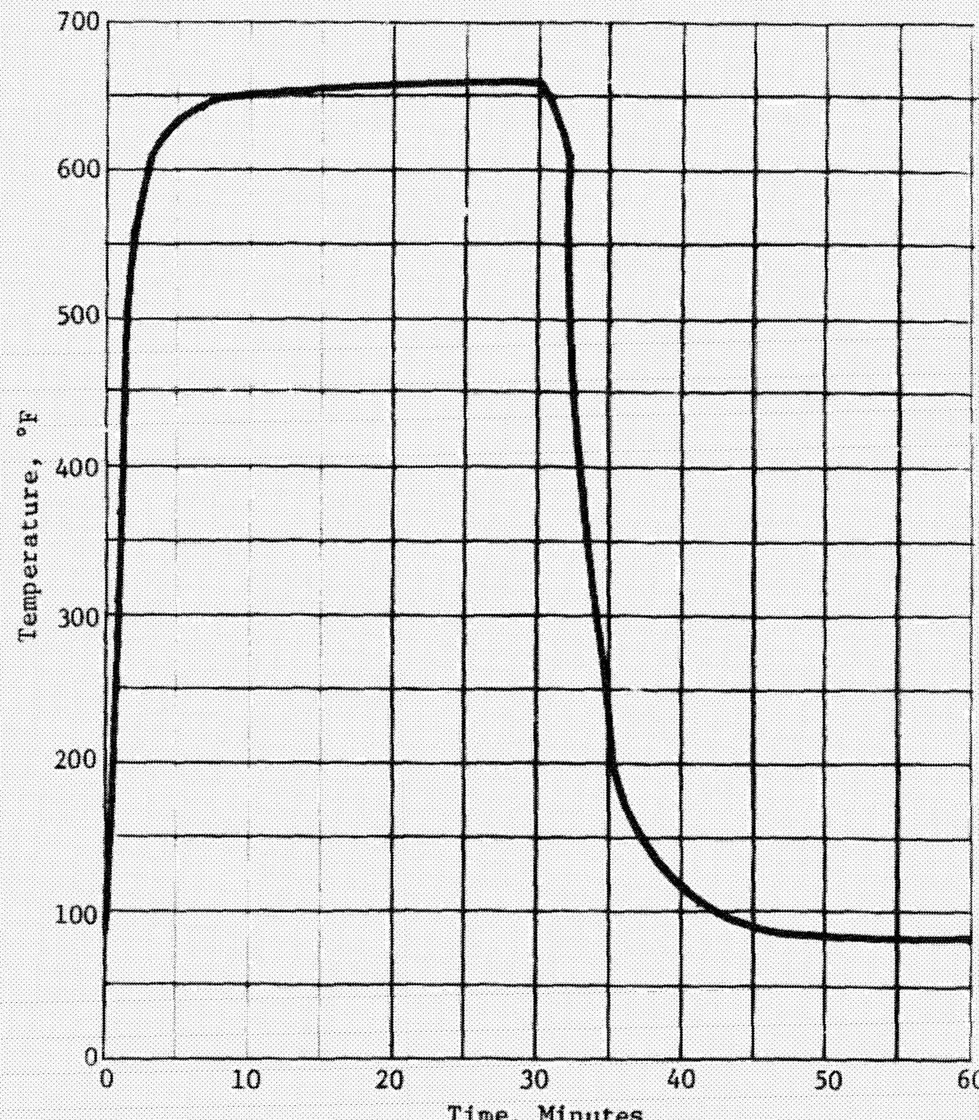


Fig. II-5 Temperature vs Time Profile of 650°F Insulation Materials During Cycle in Hydrogen Soak Test

Fig. II-6 Tensile Test and Shear Test Setup for High Temperature

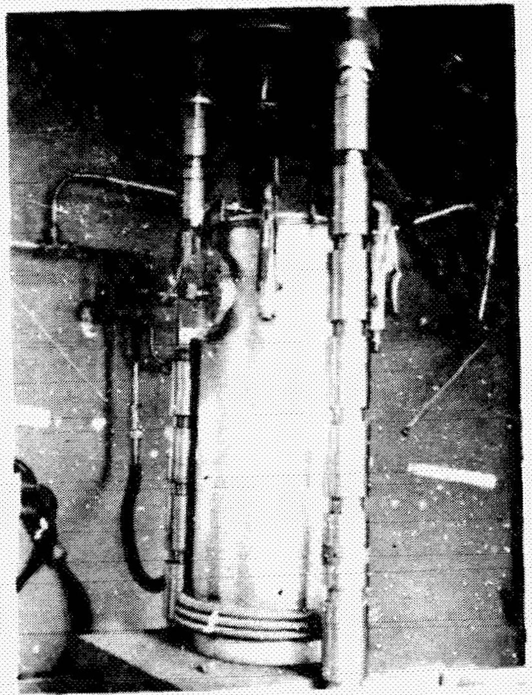


Fig. II-7 Tensile Test and Shear Test Setup for -423°F

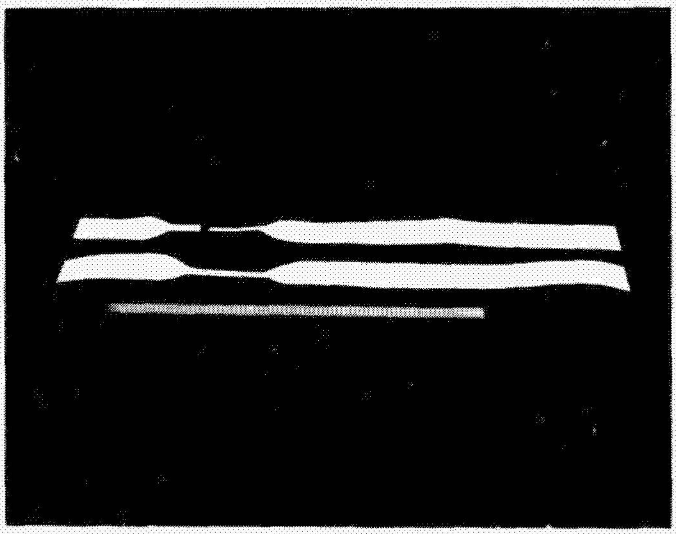


Fig. II-8 Tensile Test Specimens before and after Test at High Temperature

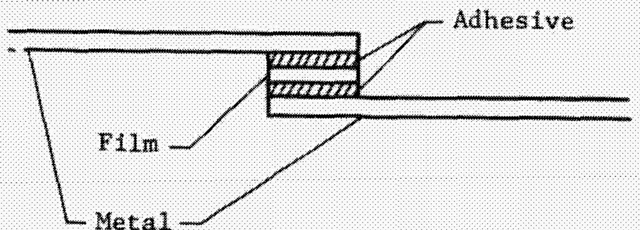


Fig. II-9 Fabrication of Lap Shear Specimen

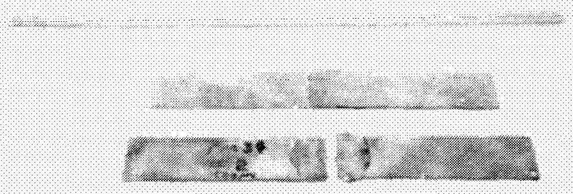


Fig. II-10 Shear Test Specimen before and after Test

3. EVALUATION OF FACESHEET AND CORE MATERIAL

The initial literature survey and our previous experience on related insulation programs indicated that the following film and core materials should be evaluated.

Material	Insulation System Application	Manufacturer
Teflon FEP Film	350°F	DuPont
Mylar Film	350°F	DuPont
Kapton Film	350/650°F	DuPont
Nomex Paper	350°F	DuPont
Nylon 6 Film	350°F	Allied Chemical

The properties determined for the above materials are summarized in Table II-1.

Screening Results

From the results of the DSC tests, the low-temperature materials all appeared to be acceptable up to 350°F. The high-temperature material, Kapton, was acceptable up to 650°F.

The Kapton, Teflon, Nomex, and nylon materials successfully completed the 200-hr hydrogen compatibility cycle test at 350°F. The mylar, however, was brittle and broke into small pieces during handling. Additional testing of Mylar was conducted in air in a 350°F oven; this Mylar also became brittle. Vendor literature indicated that Mylar is susceptible to heat-aging.

The 650°F insulation material, Kapton, changed color and became more brittle as a result of the 200-hr hydrogen compatibility cycle test at 650°F. A review of vendor literature also indicated that Kapton is susceptible to heat-aging. Data points from the vendor literature indicate that the time required to reduce the ultimate elongation from 70% to 1% is 1 yr at 660°F in helium, and 8 yr at 482°F in air. No life expectancy for Kapton at 350°F in air or helium is listed beyond the values quoted, although the data shows that the life expectancy increases significantly as the temperature decreases.

Table II-1 Properties of Films and Core Material

	Teflon FEP	Mylar (Type A)	Kapton (Type H)	Nomex (Type 412 Paper)	Nylon 6
MANUFACTURERS DATA					
Normal Temperature Range, °F	-400 to 392	-94 to 302	-322 to 392	0 to 437	-100 to 400
Reduced Properties Temperature Range	-435 to 500	-423 to 392	-452 to 752	To 600	
Melting Point, °F	500 to 535	482	None - Decomposes to a gas	None - Decomposes to a gas	390 to 425
Ultimate Tensile Strength, psi	3,000	25,000	25,000	10,000	12,000
Ultimate Elongation, %	300	125	70	15	400
Tensile Modulus at 70°F	70,000	550,000	430,000		
Specific Gravity	2.15	1.40	1.42	0.8-1.	1.13
TEST DATA					
Temperature at Which DSC Indicated Material Property Change, °F	478	441	No Change to 932	745	378
Ultimate Tensile Strength at 70°F, psi	3,1[illegible]	18,800	26,800	7,083	6,800
Ultimate Elongation at 70°F, %	320	40	18	2.7	290
Tensile Modulus at 70°F, psi	44,000	210,000	201,000	219,000	101,000
Ultimate Tensile Strength at 350°F, psi	464	4,160		8,000	2,700
Ultimate Elongation at 350°F, %	126	42		6	70
Tensile Modulus at 350°F, psi	1,822	18,800		140,000	6,300
Ultimate Tensile Strength at 650°F, psi	N/A	N/A	9,360	N/A	N/A
Ultimate Elongation at 650°F, %	N/A	N/A	42.6	N/A	N/A
Tensile Modulus at 650°F, psi	N/A	N/A	74,000	N/A	N/A
Ultimate Tensile Strength at -423°F, psi	16,600	23,000	45[illegible]00	17,400	26,000
Ultimate Elongation at -423°F, %	2.5 to 2.9	1	2.3 to 7.0	0.26 to 0.28	0.15 to 1.15
Tensile Modulus at -423°F, psi	53[illegible],000	960,000	660,000		700,000
Ultimate Tensile Strength after H_2 Thermal Cycle, psi	2,200	Shattered Into Small Pieces	4,160	8,760	12,300
Ultimate Elongation after H_2 Thermal Cycle, %	276		4.1	4.2	20
Tensile Modulus after H_2 Thermal Cycle, psi	13,000		103,000	260,000	140,000
Coefficient of Thermal Expansion to -323°F	3.9×10^{-5} in./in.-°F	0.6×10^{-5} in./in.-°F	0.85×10^{-5} in./in.-°F	0.85×10^{-5} in./in.-°F	2.4×10^{-5} in./in.-°F

The ultimate tensile strength, ultimate elongation, and tensile modulus of the films and core materials were obtained at 70°F, at 350°F for all materials, also at 650°F for Kapton, at 70°F after the 200-hr hydrogen compatibility test, and at -423°F. Table II-1 shows the lowest value of each property obtained from three specimens.

A visual observation of the failed specimens, made after obtaining the tensile properties at -423°F, showed that--

1) Kapton had the most permanent elongation. Both halves of the specimen were intact when removed from the cryostat test fixture.

2) Teflon exhibited some elongation, although less than Kapton. The Teflon specimen was also removed from the test fixture with both halves of the specimen intact.

3) Nomex exhibited negligible permanent elongation. This specimen was also removed from the cryostat test fixture with both halves intact.

4) Mylar exhibited some elongation. However, only one of the specimens was removed partially intact. On the other two specimens, only the aluminum end tabs were recovered because the Mylar had shattered.

5) Nylon exhibited some ultimate elongation. However, none of the three test specimens were recovered. Only the aluminum end tabs remained.

An infrared analysis of the Kapton film was performed to compare the material received from the vendor with the material that underwent the 200-hr hydrogen soak test. Visually, the color of the films ranged from yellow to dark brown. The infrared analysis indicated that the Kapton film contained aromatic anhydrides and aromatic amines. The main difference in the spectra of the films was in the intensity of the N-H absorption band, which increased greatly from the as-received yellow film to the dark brown film that had been thermally cycled.

A similar infrared analysis was performed on the Nylon and Teflon film. No detectable difference could be ascertained in the material that had undergone the 200-hr hydrogen cycle test. Since Nomex is not transparent, the IR analysis was not accomplished.

The most significant loads on the core and facesheet material are expected to be thermally induced. Therefore, the coefficient of thermal expansion (contraction) was measured for these materials from 70°F to -323°F.

Figure II-11 shows the test equipment used to obtain the coefficient of thermal expansion (COTE). Figure II-12 is a closeup photo of a specimen inside the transparent dewar. Note that the differential between ambient and the LN_2 condition appears on a photogrid pattern. The COTE was calculated directly from these photographs. COTE values for each material are shown in Table II-1.

2. Film Material Selection

Based on the results of our screening tests, we ranked the candidate core and facesheet materials as follows:

1) Mylar - Unacceptable due to a heat-aging problem at 350°F;

2) Nylon - Unacceptable at -423°F;

3) Nomex - This film would probably be acceptable as a core material for the 350°F insulation. It did not have as desirable an ultimate elongation at -423°F as Kapton. According to vendor literature, the Kapton has a higher modulus of elasticity than Nomex although our tests did not seem to confirm this.

4) Teflon - This film had all the desirable properties for a facesheet material. Its low yield strength at elevated temperatures is very desirable for facesheet dimpling (see the discussion in Chapter III). The one area where it is less desirable than Kapton is its specific gravity: the Teflon facesheet represents approximately 7% of the total weight of the insulation system. Because of its higher density and low modulus of elasticity, Teflon is unacceptable as a core material. Teflon is available with etched surfaces, which permits it to be bonded with a variety of adhesives.

5) Kapton - This polyimide film was the most desirable of all the films tested for use as a core material. However, because of its high yield strength at elevated temperatures, it is more difficult to dimple, and less desirable as a facesheet material. In addition, Kapton is somewhat sensitive to notches and has little resistance to the propagation of tears.

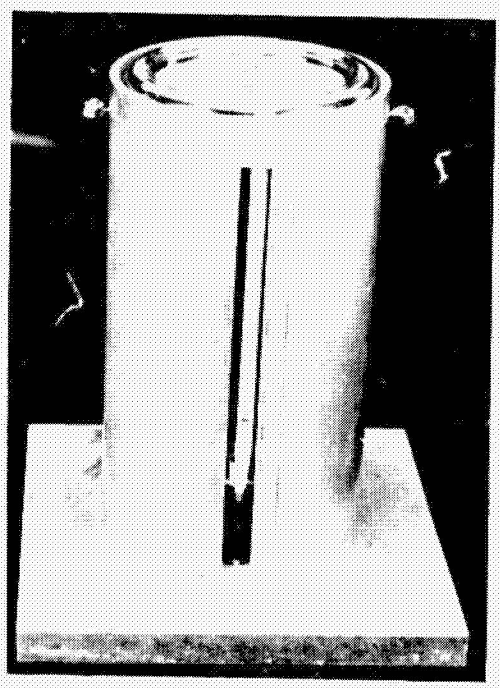


Fig. II-11 Thermal Expansion Test Fixture

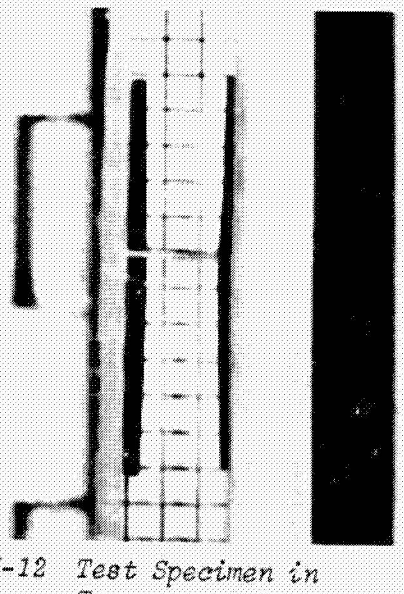


Fig. II-12 Test Specimen in Transparent Dewar

For the 350°F insulation system, we selected Kapton film as the core material and Teflon film as the facesheet material.

Kapton film was selected for both the facesheet and core for the 650°F insulation system. Kapton exhibits all the desirable properties for a core material, except high tear resistance.

C. ADHESIVES EVALUATION

Based on literature surveys and our experience on . ed programs, we selected a total of 21 adhesives for evaluation e 350°F and 650°F insulation systems. These adhesives are listed in Table II-2.

The purpose of these adhesives evaluation tests was to screen out materials with undesirable properties for the insulation system. Several different adhesive systems passed these initial screening tests but were found unacceptable for actual use during subsequent evaluations.

Table II-2 Candidate Adhesives Evaluated

Name	Insulation System		Manufacturer
	Type	Application	
Thermadite 17	Polyimide	Both	Whittaker Corp.
FM-34	Polyimide (film)	650°F	American Cyanamid
BR-34	Polyimide	650°F	American Cyanamid
7344/7119	2-Part Epoxy	350°F	Crest Products
3170/7133	2-Part Modified Epoxy	350°F	Crest Products
109-LM-52	2-Part Epoxy	350°F	Leffingwell Chemical
109-LZ	2-Part Epoxy	350°F	Leffingwell Chemical
211A/LZ	2-Part Epoxy	350°F	Leffingwell Chemical
3M-3558	2-Part Epoxy	350°F	3M
HT-424	2-Part Epoxy Phenolic	350°F	American Cyanamid
H-21	2-Part Epoxy	350°F	Epoxy Tech.
H-61	2-Part Epoxy	350°F	Epoxy Tech.
H-74	2-Part Epoxy	350°F	Epoxy Tech.
H-75	2-Part Epoxy	350°F	Epoxy Tech.
WD-5332	1-Part Epoxy	350°F	American Cyanamid
Epibond 8510	2-Part Epoxy	350°F	Furane Plastics, Inc.
RT-560	2-Part Methyl-Phenyl Silicone	Both	General Electric
RT-156	1-Part Silicone	Both	General Electric
RT-583	2-Part Dimethyl Silicone	350°F	Dow Corning
RT-589	2-Part Dimethyl Silicone	350°F	Dow Corning
RT-96-0[illegible]2	2-Part Methyl-Phenyl Silicone	350°F	Dow Corning

Although the exact criteria for selecting adhesives for the three applications in the internal insulation system were not well known, the following characteristics are either obviously required or were believed to be desirable.

1) Compatibility with hydrogen gas at the upper temperature condition for long periods;

2) Compatibility with liquid hydrogen;

3) High strength at high and cryogenic temperatures;

4) Flexibility at high and low temperatures;

5) Low coefficient of thermal expansion;

6) Low density;

7) Low thermal conductivity.

1. Screening Results

The results of screening tests on the adhesive materials are summarized in Table II-3. The shear stress shown is the lowest value obtained for three lap shear specimens. The lap shear specimens for the 350°F adhesives were made with 2219 aluminum alloy, but those for the 650°F adhesives were made with 6Al-4V ANL titanium and with Inconel 718.

Not all screening tests were performed for each adhesive. Some adhesives were only briefly considered; others were evaluated extensively. All adhesives were used in accordance with the manufacturers' recommendations, except where noted in the discussion of a particular adhesive.

In addition to tests reported in Table II-3, we devised a test to subject a number of the candidate 350°F adhesives to direct exposure to liquid hydrogen. Several strips of adhesive, each ½ in. wide and ¼ in. thick, were applied to an aluminum dome (see Fig. II-13), and the dome was pressurized to 45 psig ten times, with liquid hydrogen in contact with the adhesives. After this, the dome was cycled from 350°F to -420°F three additional times. This test subjected the adhesives to the maximum expected loads due to differential thermal contraction and to the maximum strain in the wall (or dome) material due to internal pressure. Results of the dome strip tests are summarized in Table II-4. The candidate adhesives were judged to have successfully withstood the test if no debonding or cracking occurred.

Table II-3 Summary of Properties of Adhesive Materials

	RTV-[illegible]6	RTV-560	BR-34	Thermadite -17	RTV-581	RTV-58[illegible]	96-05[illegible]	FM-34	[illegible]	1170 [illegible]	10[illegible] IBR-[illegible]
MANUFACTURER PUBLISHED DATA											
Recommended Temperature Range, °F	-85 to 600	-175 to 500		To 500	-30 to 400	-30 to 400	-175 to 400	-320 to 1000	-423 to 350	-423 t[illegible] 350	-423 t[illegible]
Specific Gravity	1.11	1.42			[illegible].17	1.[illegible]0	1.18			1.02	1
Ultimate Shear Stress at Room Temperature, psi					250	500		2800	[illegible]	3000	[illegible]600
Ultimate Shear Stress at Other Temperatures, psi						2096 at -320°F		2600 at 180°F 4200 at 320°F	1200 a 180°F, 3580 at -320°F 2060 at -423°F	500 at 180°F 2800 at 320°F 2990 at -423°F	
TEST DATA											
Ultimate Shear Stress at Room Temperature, psi	96	288	2060	1150	*	170*	80	1800	1082		1081
Ultimate Shear Stress at 350°F, psi	95.9	37.4	1490	1288	24.2	17.	1773	240	250		2[illegible]
Ultimate Shear Stress at -423°F, psi	825	1098	2660	2140		60	1515	2320	990		818
Ultimate Shear Stress at Room Temperature after H_2 Cycling (200 cycles) to 350°F, psi	230	222	1675	415	108	156	136	1620	140		808
Ultimate Shear Stress at Room Temperature after H_2 Cycling (200 cycles) to 650°F, psi		88 (75 cycles)	680	180				1070			
Ultimate Shear Stress at 650°F, psi			226	114				240			
Thermal Stability by Differential Scanning Calorimeter 100 - 350°F									No Reaction	Unacceptable	No Reaction
100 - 650°F			No Reaction	No Reaction				No Reaction			

	[illegible]-17	[illegible]A-17	HT [illegible]	IM-[illegible]	Epibond [illegible]	[illegible]D [illegible]	H-[illegible]	H-[illegible]	H-[illegible]	H-[illegible]
MANUFACTURER PUBLISHED DATA										
Recommended Temperature Range, °F		-423 to [illegible]	-400 t[illegible]		[illegible] 300	[illegible] to [illegible]	[illegible]	T[illegible] 400	T[illegible]	
Specific Gravity						1.				
Ultimate Shear Stress at Room Temperature, psi			21[illegible]	[illegible]	[illegible]	1800	1000		[illegible]	
Ultimate Shear Stress at Other Temperatures, psi	780 at 1[illegible]0°F	[illegible] at 1[illegible]0°F [illegible] at -320°F 600 at -423°F	2500 at 1[illegible]°F	[illegible] at 1[illegible]0°F [illegible] at -3[illegible]°F	[illegible] at 1[illegible]0°F	[illegible] a 1[illegible]0°F				
TEST DATA										
Ultimate Shear Stress at Room Temperature, psi	[illegible]	[illegible]	2[illegible]	1[illegible]	18[illegible]	[illegible]650	1616	[illegible]	110	[illegible]
Ultimate Shear Stress at 350°F, psi	1180	156[illegible]	1650	6[illegible]8	[illegible]	1[illegible]1	6[illegible]	246	4[illegible]	480
Ultimate Shear Stress at -423°F, psi	1560	162[illegible]	180[illegible]	1310	1[illegible]00	>1800		1070	[illegible]10	1560
Ultimate Shear Stress at Room Temperature after H_2 Cycling (200 cycles) to 350°F, psi	2110	[illegible]161	144[illegible]	[illegible]60	23[illegible]	116	1260	[illegible]	[illegible]	1700
Ultimate Shear Stress at Room Temperature after H_2 Cycling (200 cycles) to 650°F, psi										
Ultimate Shear Stress at 650°F, psi										
Thermal Stability by Differential Scanning Calorimeter 100 - 350°F										
100 - 650°F										

*Failed when strip or adhesive, bonded to aluminum dome, was submerged in liquid hydrogen.
†Pin hole in aluminum strip failed, all specimens.

Fig. II-13 350°F Adhesives Bonded to Aluminum Dome

Table II-4 Dome Strip Test Results at 350°F

Adhesive	Type	Test Results
RTV-583	Dimethyl Silicone	Failed
RTV-589	Dimethyl Silicone	Failed
3M-3558	Epoxy	Failed
Epibond 8510	Epoxy	Successful
H-21	Epoxy	Successful
H-61	Epoxy	Successful
H-74	Epoxy	Successful
H-75	Epoxy	Successful
109/LZ	Epoxy	Successful
211/LZ	Epoxy	Successful
WD5332	Epoxy	Successful
RTV-560	Methyl Phenyl Silicone	Successful
RTV-96-052	Methyl Phenyl Silicone	Successful
RTV-156	One Part Silicone	Successful

2. 350°F Adhesives Selection

The Crest 3170/7133 adhesive proved unacceptable at 350° in DSC tests. Contact with the vendor revealed that one of the constituents would melt at approximately 300°F, as our DSC tests had indicated. The test results for the Crest 7344/7119 were not as favorable as for the Lefkoweld 109/LM-52. However, because of its high viscosity, the 109/LM-52 was difficult to use for bonding core node points and for bonding the core to the facesheet. Thermadite-17 was primarily evaluated for use with the 650°F system, but was also considered for bonding the core node points for the 350°F system. This adhesive was developed as a Kapton-to-Kapton adhesive, and its disadvantage is that it requires heat and pressure to cure.

Based on these results, we initially selected the following adhesives for the 350°F insulation system:

1) Thermadite 17 for bonding the core node points;

2) Lefkoweld 109/LM-52 for bonding the facesheet to the core and the core to the metal.

These adhesives were incorporated into system tests, as described in Appendix A, but were eliminated as a result of these tests. Lefkoweld 2il A/LZ was evaluated further and was found to have favorable properties, but it was very difficult to use because of its very high viscosity. At this point, the silicone family of adhesives was investigated.

Although RTV (room-temperature vulcanizing) silicones are not normally considered good structural adhesives, they have the following desirable properties for application to the internal insulation:

1) Elastic behavior to -75°F or -175°F, depending on whether a dimethyl or methyl-phenyl type adhesive is used;

2) Flexibility at room temperature, which allows normal handling without damaging cells;

3) Minimum stress created in cooldown from room temperature to -175°F:

4) Satisfactory shear strength at cryogenic temperatures;

5) Satisfactory bonding strength at 350°F;

6) Room-temperature curing, which simplifies fabrication and repair;

7) Desirable flow and wetting characteristics, resulting in a fillet that fills voids and produces leak-free bonds for core/facesheet bonding and core/metal bonding.

These properties made the silicone adhesives candidates for use in the 350°F materials system, and they were evaluated further in systems tests. Ultimately, they were also evaluated for the 650°F materials system, although the polyimide adhesives had initially appeared to be superior for that application. In testing the silicone adhesives, we used several of the recommended primers. These primers all proved satisfactory, and primer selection was based on fabrication considerations, as discussed in Chapter III.

Five different silicone adhesives were initially evaluated for use in the 350°F insulation system. The RTV-583 and RTV-589 adhesives were eliminated in the dome strip tests. RTV-96-052 is primarily a potting compound, and was considered less adaptable for use as an adhesive than RTV-560 and RTV-156. RTV-560 and RTV-156 adhesives were selected for further evaluation in the 350°F insulation system tests, as described in Chapter IV and Appendix A.

3. Core Node Adhesive Selection (350°F and 650°F)

Thermadite-17, a polyimide adhesive, was initially selected as the node adhesive for fabricating the Kapton core. This material is compatible with both the 350°F and 650°F operating temperatures. A persistent problem was experienced with the core assembly, however, due to the rigidity of the polyimide adhesive. The rigid bond lines created points of stress concentration in the Kapton film, and as a consequence, the core frequently became damaged from ordinary handling during subsequent fabrication steps.

To overcome this problem, we evaluated silicone adhesives for bonding the core nodes. Both RTV-560, the two-part methyl-phenyl silicone, and RTV-156, a one-part, self-priming adhesive were tried. Of these, the RTV-156 adhesive proved to be an excellent choice. This adhesive has a high peel strength and high-temperature capability, requires no primer, and has the proper consistency to be stenciled on the Kapton ribbons. In addition, the RTV-156/Kapton core bond is very flexible at room temperature, which permits normal handling without tearing the core ribbons. On the other hand, because it is cured by exposure to moisture in the air, it has a relatively short working time. Nevertheless, an adequate working time can be achieved by purging the stencil box with nitrogen.

The peel strength of the RTV-156 was measured at room temperature in the "T" configuration. This property was found to depend on the thickness of the adhesive layer between the ribbons. At below 0.005 in., the peel strength was 6 lb/in. or less. At an applied thickness of 0.012 to 0.015 in., the peel strength varied from 15 to 23 lb/in. The strength increased greatly as the thickness was increased further. However, 0.015 in. is near the maximum thickness that can be used without excessive spreading of the node bonds when they are compressed for bonding.

At -320°F the peel strength of the RTV-156 was equal to or greater than that at room temperature.

Some difficulties were initially experienced in obtaining consistent results with the RTV-156. These problems and their solutions are discussed in Chapter III.

RTV-560 would also be suitable as a core node adhesive, but it has a peel strength of only 1.5 to 3 lb/in. It also is more difficult to use because a primer is required for bonding it to Kapton. As a result, the RTV-156 adhesive was selected for bonding the core nodes in the 350°F system, and was used in the insulation system tests described in Chapter IV. (RTV-156 was later determined to be acceptable for the 650°F system as well, and was evaluated in system tests.)

4. 650°F Adhesives Selection

Polyimide adhesives have excellent properties at 650°F. The original adhesives selected for the 650°F system were BR-34, for bonding the facesheet to the core and the core to the tank, and Thermalite-17 for the core node bonds.

The manufacturer of BR-34 recommends that a coating of this adhesive, 1 to 2 mils thick and properly cured, be used as a primer. Published data were reviewed and several manufacturers and users were contacted to determine whether other primers were available for this application No promising alternatives were found.

To evaluate the effectiveness of the BR-34 priming system for use with BR-34 adhesive, we fabricated 24 lap shear specimens using 0.050-in. Inconel 718 and BR-34, with a tab of Kapton film in the shear bond (see Fig. II-9). Half of the Inconel 718 tabs were primed with a thin coating of BR-34 after being cleaned. The primer was cured by air-drying for 30 minutes, oven-drying at 220°F for 30 minutes, and curing for 45 minutes at 410°F. The other tabs were not primed.

Half of the primed specimens and half of the unprimed specimens were then subjected to 75 thermal cycles in a hydrogen environment. A temperature of 630°F or higher was reached in at least 38 of these cycles. Because of a heater malfunction in the test apparatus, the maximum temperature reached during the other cycles varied between 630°F and 470°F. The temperature decreased to 230°F or below during each cycle.

These temperatures were measured near the center of the specimen container, which was cycled in and out of a heated salt bath. Specimens nearer the wall of the container experienced both higher and lower temperature extremes.

All the specimens were then tensile-tested. The average ultimate shear stress for specimens that had not been thermally cycled was 2498 psi for the primed specimens and 2908 psi for the unprimed ones. For specimens tested after thermal cycling, the average ultimate shear stress for the primed specimens was 2403 psi, compared with 2477 psi for the unprimed specimens. For the wall bonding application for the internal insulation system, these strengths are far more than adequate. And since the primary benefit from priming the surfaces would be to protect the cleaned metal surface, this priming procedure would be recommended for systems using the BR-34 adhesive.

Peel specimens were also fabricated of Kapton bonded to Inconel 718 with BR-34. In specimens that were thermally cycled, as well as in those that were not, the Kapton failed with a negligibly small force due to the stress concentrations that occurred at the edge of the rigid bond.

The most important disadvantage of the polyimide adhesives proved to be the rigidity of the core node bonds. In addition, these adhesives are not 100% reactive, but contain solvents, some of which were released as the adhesive cured. This resulted in porosity and voids in the bonds, with consequent cell-to-cell leakage.

Although silicone adhesives were originally considered only for the 350°F system, we conducted several tests to evaluate their suitability for use at 650°F.

Lap shear specimens and peel test specimens were fabricated from 0.050-in. Inconel 718 to evaluate the RTV-560 core-to-metal bond at 650°F. These specimens were fabricated by bonding a piece of Kapton between Inconel tabs using RTV-560 adhesive. Peel specimens were fabricated by bonding 1-in.-wide strips of 5-mil Kapton film to 1-in. by 8-in. Inconel strips. The specimens were prepared with two different primers, either GE 4004 or Dow Corning DC-1200, to evaluate the effect of primers on the core-to-metal bond.

Six specimens prepared with each primer were subjected to 75 thermal cycles in a hydrogen environment, with the same conditions described above for the BR-34 primer. The RTV-560 specimens changed from red to a very dark brown or black color following the hydrogen cycles. Small samples of this adhesive that had been previously exposed to a temperature of 650°F in air for 72 hr did not exhibit such a change in color.

After undergoing the thermal cycle tests, the lap shear and peel test specimens were tested at room temperature. Specimens that had been fabricated at the same time, but not thermally cycled, were also tested for comparison. The peel strength was measured in the 180°F test configuration. The results of these tests are given in Tables II-5 and II-6. Because a minimum of five specimens were tested for each configuration, only average values are reported.

The RTV-560 lost from 57 to 69% of its original strength in shear, but still retained excellent elastomeric qualities. In peel, the loss of strength was only 22 to 30%. We concluded that the adhesive had retained sufficient strength to meet the requirements for the internal insulation system. In subsequent system tests, described in Chapter IV, the RTV-560 adhesive was proven to be an excellent choice for the 650°F system.

Although silicone adhesives were chosen for the final series of system tests, BR-34 was not determined to be unacceptable.

Table II-5 Test Results for RTV-560 Silicone Adhesive/Kapton/Inconel Lap Shear Specimens

Primer	Average Ultimate Shear Stress, psi	
	As Fabricated (not thermally cycled)	After 75 Thermal Cycles in Hydrogen
Dow Corning 1200	317	104
General Electric SS-4004	336	136

Table II-6 Test Results for RTV-560 Silicone Adhesive/Kapton/Inconel Peel Specimens

Primer	Average Ultimate Peel Strength, lb/in.	
	As Fabricated (not thermally cycled)	After 75 Thermal Cycles, in Hydrogen
Dow Corning 1200	2.7	2.1
General Electric SS-4004	2.7	1.9

The RTV-156 one-part adhesive used for node bonding was also subjected to the high-temperature hydrogen cycle exposure. Afterward, the average peel strength from 10 measurements was only 1.1 lb/in. Since the primary requirement for high peel strength is to prevent

damage during fabrication and handling, this value is acceptable after the panels are installed. When used in 650°F system tests, no comparable degradation of the RTV-156 bonds occurred.

FILLER MATERIAL EVALUATION

The filler material used to pack the core cells can be any suitable material that will control convection. Owens-Corning PF-105-700 and PF-105-450 fiberglass proved very satisfactory as filler materials. Since the filler material selected does not affect other insulation materials, we did not evaluate other filler materials in detail.

Both opacified and nonopacified fiberglass batting were used in fabricating insulation specimens. The opacified batting material was made on special order by Owens Corning Fiberglass, using a process to blow aluminum powder onto the glass fibers during the felting operation. This opacified fiberglass was effective in reducing radiant heat transfer and would be desirable for high-temperature applications if effective insulation (rather than survival) were required at high wall temperatures.

E. SELECTED MATERIALS

The materials that were finally selected for the 350°F and 650°F insulation systems are listed in Table II-7. The selected materials systems are designated 350-III and 650-II. Chapter IV discusses test results for insulation systems made with these materials.

The material systems that were originally evaluated in the system tests are listed in Table II-8, and are designated 350-I, 350-II, and 650-I. Test results for these materials are discussed in Appendix A.

The 650-I materials were evaluated with 6Aℓ-4V ANL titanium alloy as the tank metal, and the 650-II materials were evaluated with Inconel 718. All three 350°F materials were evaluated with 2219 aluminum alloy.

Table II-7 Selected Insulation Materials

	350-III System		650-II System	
Component	Material	Manufacturer	Material	Manufacturer
Core Ribbon	5-mil Kapton film (Type H)	Dupont	5-mil Kapton Film (Type H)	Dupont
Facesheet	2-mil Teflon FEP Film (etched both sides)	Dupont	1-mil Kapton Film (Type H)	Dupont
Core Node Adhesive	RTV-156	General Electric	RTV-156	General Electric
Core-to-Facesheet Adhesive	RTV-560 with 0.25% DTD*	General Electric	RTV-560 with 0.25% DTD	General Electric
Core-to-Metal Adhesive (and grout compound)	RTV-560 with 0.25% DTD, 5% RTV-9811, and 1% Cab-O-Sil	General Electric Cabot	RTV-560 with 0.25% DTD, 5% RTV-9811, and 1% Cab-O-Sil	General Electric
Primer on Core, Facesheet, and Metal	DC-1200	Dow Corning	DC-1200	Dow Corning
Filler Material	PF-105-450 Fiberglass Batting	Owens-Corning	PF-105-700T Opacified† Fiberglass Batting	Owens-Corning

*DTD = dimethyl tin dilaurate.

†Opacified or nonopacified fiberglass would be selected on the basis of system requirements.

Table II-8 Candidate Materials Systems Tested

Component	350-I System		350-II System		650-I System	
	Material	Manufacturer	Material	Manufacturer	Material	Manufacturer
Core Ribbon	5-mil Kapton Film (Type H)	Dupont	5-mil Kapton Film (Type H)	Dupont	5-mil Kapton Film (Type H)	Dupont
Facesheet	2-mil Teflon FEP Film (etched both sides)	Dupont	2-mil Teflon FEP Film (etched both sides)	Dupont	1-mil Kapton Film (Type H)	Dupont
Core Node Adhesive	Thermadite-17	Whittaker	Thermadite-17	Whittaker	Thermadite-17	Whittaker
Core-to-Facesheet Adhesive	Lefkoweld 109/LM-52	Leffingwell	RTV-560	General Electric	BR-34	American Cyanamid
Core-to-Metal Adhesive	Lefkoweld 109/LM-52	Leffingwell	RTV-156	General Electric	BR-34	American Cyanamid
Primers on Metal on Core and Facesheet	None None None		None 1200 1200	 Dow Corning Dow Corning	Thinned BR-34 None None	American Cyanamid
Filler Material	PF-105-700T Fiberglass (Opacified)	Owens-Corning	PF-105-700T Fiberglass (Opacified)	Owens-Corning	PF-105-700T Fiberglass (Opacified)	Owens-Corning

The estimated total weight per square foot for the selected insulation materials systems is given in Tabl II-9. These values are based on an insulation thickness of 1 in., and have been estimated using the design, fabrication, and installation concepts presented in Chapter III. A cell size of 2.04 in. (height) by 1.73 in. (average width) was chosen, and the weights shown in the table have actually been achieved for prefabricated panels using these cell dimensions. A stencilled adhesive pattern corresponding to the core pattern has been assumed for installation, with the adhesive strips ¼ in. wide and 1/16 in. thick.

The weight of the system will decrease as the cell size is increased, so the weight estimates presented here are believed to be achievable, and probably conservative.

Table II-9 Estimated Total Weight of Selected Insulation Systems per Square Foot (Based on 1 in. Thickness of Insulation)

Component	350-III System		650-II System	
	Material	Weight, lb/ft^2	Material	Weight, lb/ft^2
Core Ribbon	0.005-in. Kapton	0.0595	0.005-in. Kapton	0.0595
Node Bond Adhesive	RTV-156	0.0062	RTV-156	0.0062
Facesheet	0.002-in. Teflon FEP	0.0224	0.001-in. Kapton	0.0074
Facesheet-to-core Adhesive	RTV-560	0.0334	RTV-560	0.0334
Filler	PF-105-item	0.0530	PF-105-item	0.0530
Primer for Prefabricated Panels	DC-1200	0.0066	DC-1200	0.0066
Primer for Tank Wall	DC-1200	0.0030	DC-1200	0.0030
Installation Adhesive	RTV-560	0.1620	RTV-560	0.1620
Total Weight		0.3461		0.3311

III. DESIGN AND FABRICATION

A. DESIGN ANALYSIS

The capillary internal insulation concept relies on the surface tension of the cryogen to maintain a gas layer adjacent to the tank wall. The basic design concept is illustrated in Fig. III-1. The insulation consists of a cellular core attached to the tank wall and a perforated facesheet attached to the inner surface of the core. The facesheet has one capillary opening per cell. The cells can be filled with any suitable material, such as opaque fibrous batting, to control convection and radiation. Each cell acts independently.

A stable liquid/gas interface forms at each capillary opening. This prevents the liquid from entering the cell and positions a gas layer between the liquid and the tank wall. Because pressure is equalized across this interface (except for a very small capillary pressure difference) the system is essentially free of pressure-induced structural loading. Consequently, the insulation system can be designed with a lightweight structure and a wide range of materials. By incorporating excess facesheet and core material to accommodate thermal contraction, the design can be made relatively free of thermally-induced stresses.

1. Capillary and Thermodynamic Considerations

The stability of the capillary liquid/gas interface is determined by the same physical relationships that apply to the liquid/gas interface formed when a liquid is positioned at the top of an inverted cylinder closed at one end (i.e., the familiar soda straw experiment). The stability of the capillary interface is determined by a critical Bond number (Bo) relationship (a ratio of gravity forces to surface tension forces) that establishes the maximum size of the capillary hole. For this situation, the limit of stability for a perfectly wetting liquid is determined by

$$Bo = \rho \frac{g}{g_c} \frac{d^2}{\sigma} \leq 3.37,$$

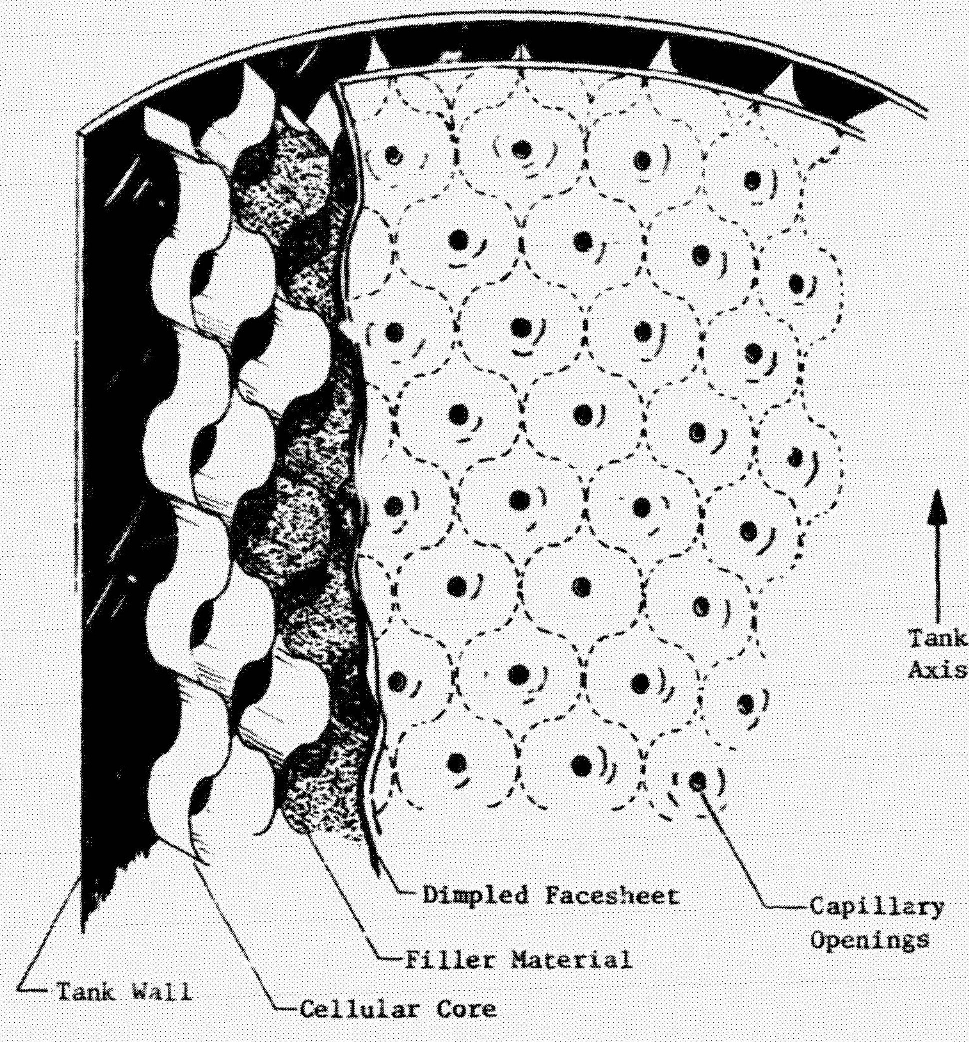


Fig. III-1 Capillary Internal Insulation Concept

where:

Bo = Bond number, dimensionless

ρ = density of liquid (minus density of gas), lb_m/ft^3

$\frac{g}{g_c}$ = ratio of acceleration or local gravitational field to gravitational constant, lb_f/lb_m

d = diameter of capillary opening, ft

σ = surface tension, lb_f/ft.

Thus, capillary hole size is limited by the relationship

$$d_{max} = \sqrt{\frac{3.37}{\rho}\frac{g_c}{g}(\sigma)}.$$

In practice, a much smaller capillary opening can be used with a greater stability margin. The lower limit of capillary hole size is determined by pressure-equalization considerations.

For space vehicle applications, three different gravitational environments exist: 1 g during fueling, 3 g during the boost phase, and zero g during flight. The maximum capillary hole diameter for each environment is given below.

For liquid hydrogen,

$\sigma = 13.05 \times 10^{-5}$ lb/ft,

$\rho = 4.43$ lb/ft^3.

For 1 g, $d_{max} = \left[\frac{(3.37)}{(4.43)}\frac{32.2}{32.2} 13.05 \times 10^{-5}\right]^{\frac{1}{2}} = 0.00992$ ft (0.119 in.)

For 3 g, $d_{max} = \left[\frac{3.37}{4.43}\frac{32.2}{3(32.2)} 13.05 \times 10^{-5}\right]^{\frac{1}{2}} = 0.00575$ ft (0.069 in.)

For 0 g, $d_{max} = \left[\frac{3.37}{4.43}\frac{(32.2)}{0(32.2)} 13.05 \times 10^{-5}\right]^{\frac{1}{2}} = \infty$

For the internal insulation system, a capillary hole size of 0.020 to 0 [illegible] 0 in. is acceptable and practical for fabrication.

The above analysis is based on a contact angle of zero for perfectly wetting liquids. The contact angle is the angle formed through the liquid at the line of contact between liquid, gas, and solid. This angle generally depends on the liquid and solid being used. Since a contact angle larger than zero would increase stability, the Bond number relationship is valid for establishing capillary criteria.

A static pressure differential across the facesheet can exist as a result of surface tension forces. For a circular opening, the maximum capillary pressure differential (ΔP) is

$$\Delta P = \sigma \left(\frac{1}{R_1} + \frac{1}{R_2}\right) = \frac{4\sigma}{d}$$

where R_1 and R_2 are the major and minor radii of curvature at any point on the static liquid/gas interface, and d is the diameter of the capillary opening. For a liquid hydrogen system and a capillary diameter of 0.035 in., the pressure differential is on the order of 0.001 psi. This capillary pressure depends on the shape of the interface, and will be a maximum when a hemispherical bubble is formed at the opening.

When a liquid cryogen first contacts the insulation, the gas in the cells cools and contracts, tending to lower the cell pressure. As a result, some liquid enters the cells and begins to vaporize because the temperature within the cells is higher than the boiling point for the cryogen. The gas that is produced then tends to pressurize the cell. As the cell pressure exceeds the local pressure in the bulk liquid, gas is discharged from the cell through the opening, forming bubbles in the liquid.

This process of gas entering the cell, vaporizing, and being discharged from the cell will continue until a state of thermal and pressure equilibrium is reached. If the tank or head pressure is increased, this sequence of events will be repeated until a new equilibrium is achieved. Similarly, if the tank pressure is decreased, gas will be discharged from the cell until the pressure is again equalized.

The pressure that can exist across the facesheet depends on the rate of change of tank pressure and on the relationship between the facesheet hole size (and the consequent flow-loss characteristic) and the cell volume. The small differential (capillary) pressure that can be supported by the surface tension of the interface can exist without any flow of liquid or gas through the facesheet opening.

2. Thermal "Stress-Free" Design Concept

Because the insulation system is pressure-equalized, it does not support steady-state liquid or pressure loads. Rather, these loads are transmitted through the columns of gas in the insulation cells directly to the tank walls. In effect, factors such as rapid pressure changes and changes in the head pressure of the liquid due to vehicle acceleration changes and propellant sloshing produce the significant loads on the insulation, but these loads are relatively minor. Because the structural load-carr[illegible] requirements are small, the insulation system can be des[illegible] to be lightweight, flexible, and relatively free of the[illegible] induced stresses.

The design approach in minimizing thermal stresses is to build in additional material, so that the members do not become loaded in tension when thermal contraction occurs. For cellular core, this is accomplished by configuring the ribbon that forms the cell walls in an S-shape. The actual length of the ribbon exceeds the straight-line distance between the node bonds by a factor more than sufficient to compensate for contraction.*

In practice, the excess ribbon length for each span is approximately 5½%. This is several times (a factor of 10 or more for Kapton) greater than the contraction of the ribbon in going from room temperature to that of the liquid hydrogen, and is near the maximum excess length that can be accommodated before cell buckling occurs.

---

*Since the segments of the ribbons joined by the node bond are common to two adjacent cells, those segments are not free to distort to accommodate thermal contraction. Therefore the node bonds are kept narrow in order to increase the portion of the ribbon that is free to distort into a new path between node points.

To compensate for the contraction of the facesheet, it is dimpled. Attempts to analyze the distortion of a facesheet bonded to a distorted cell ribbon and to determine the minimum dimpling required have been inconclusive. Our experience has shown that a total dimple depth of 0.075 in. per inch of facesheet span is sufficient to prevent tears in Teflon. And although the different rates of thermal contraction for the core material, adhesive, and facesheet result in thermal stresses, these are readily overcome by the adhesive.

3. Cell Size Considerations

The primary limitation on cell size is set by the nature of the transient pressure changes on the tank and the ability of the facesheet to momentarily withstand a pressure load. There is also a limit to the size of the opening in the facesheet, which is based on capillary stability considerations. Therefore, for a given rate of pressure change in the tank, assuming the other parameters are fixed, the rate of pressure equalization, and consequently, the maximum differential pressure across the facesheet, can be limited by limiting the cell volume.

In addition, the core material must be able to withstand the compressive load induced by a differential pressure across the facesheet, as well as other loads that may be imposed. Based on past experience, the most severe compressive loads imposed on the core will be those incurred during panel fabrication. Current fabrication methods require the core to be capable of withstanding a compressive load of 1 to 2 psi.

Other considerations affecting cell size are system weight and, possibly, cost. Since the total length of the ribbon and the number of node bonds per unit area decrease as the cell size increases, the weight of adhesive would also decrease as the cells are made larger.

The structural properties of the cellular core material used in the internal insulation system are quite unlike those for structural honeycomb sandwiches. However, the analytical approaches used for structural honeycomb were employed to gain an understanding of the factors involved in minimizing core weight.

The flatwise compressive strength of large-cell core is determined on the basis of column buckling. In column buckling the property of interest is the tensile modulus, rather than the tensile strength. If the core between two nodes is considered as a plate, the allowable buckling stress is

$$F_{CR} = KE\left(\frac{t}{b}\right)^2.$$

However, if the plate is curved, which it is when the core is expanded, then

$$F_{CR} = CE\ \frac{t}{R},$$

where:

R = radius of curvature,

K = function of Poisson's ratio,

C = empirical function determined by test,

b = length of core between nodes,

E = modulus of elasticity,

t = thickness of core ribbon

The parameter, then, that should be used to minimize the weight of the insulation system for the core material is the modulus of elasticity.

For a fixed modulus of elasticity and geometrically similar designs, it would appear that the product of ribbon thickness and total ribbon length per unit area is constant for a given compressive strength. By this argument, weight is not a function of cell size if the optimum ribbon thickness is chosen in all cases.

The more important features of our core design are the S-shaped ribbons and narrow bond lines, rather than the optimization of structural capabilities. The S-curve provides excess ribbon to prevent tension loads due to thermal contraction. Cells from 1 to 3 in. in their major dimension have been fabricated using 0.005-in. Kapton film and have exhibited sufficient compressive strength. Exact cell dimensions are set by tank size and geometry when the nested panel design concept, discussed below, is used.

B. DESIGN AND FABRICATION DEVELOPMENT

Although the insulation concept is relatively simple, we found that considerable attention--both to design and fabrication details--was essential to its successful application. Consequently, a major part of this program dealt with developing design and fabrication methods for insulating an entire large tank even though only small panels and domes were used to prove the concepts.

These efforts had three basic objectives: to obtain prefabricated insulation panels with consistently high quality; to devise techniques that would enable these prefabricated panels to be installed in the field with a minimum of effort; and to develop techniques for repairing the insulation in case it were inadvertently damaged after being installed.

The insulation specimens that were initially fabricated had a rectilinear core with straight Kapton ribbons. The rectilinear core was fabricated by applying parallel lines of adhesive to the Kapton ribbons at regular intervals and bonding the ribbons together, either in a flat pattern or in expanded form. Core bonding in a flat pattern was more efficient, but this required selecting an adhesive with the proper viscosity and using a resonable bond pressure to prevent the bond line from spreading.

With the polyimide adhesives (which required high bond pressures), it was necessary to bond the core in expanded form, as shown in Fig. III-2. With the silicone adhesives, the core was first bonded in a flat pattern (see Fig. III-3) and then expanded on a rake.

Before bonding the facesheet to the core, the core was first expanded on a contoured rake (whose shape corresponded to the installed curvature of the insulation) to produce the proper cell shape. In this way, the core would retain its expanded shape after the facesheet was bonded to it.

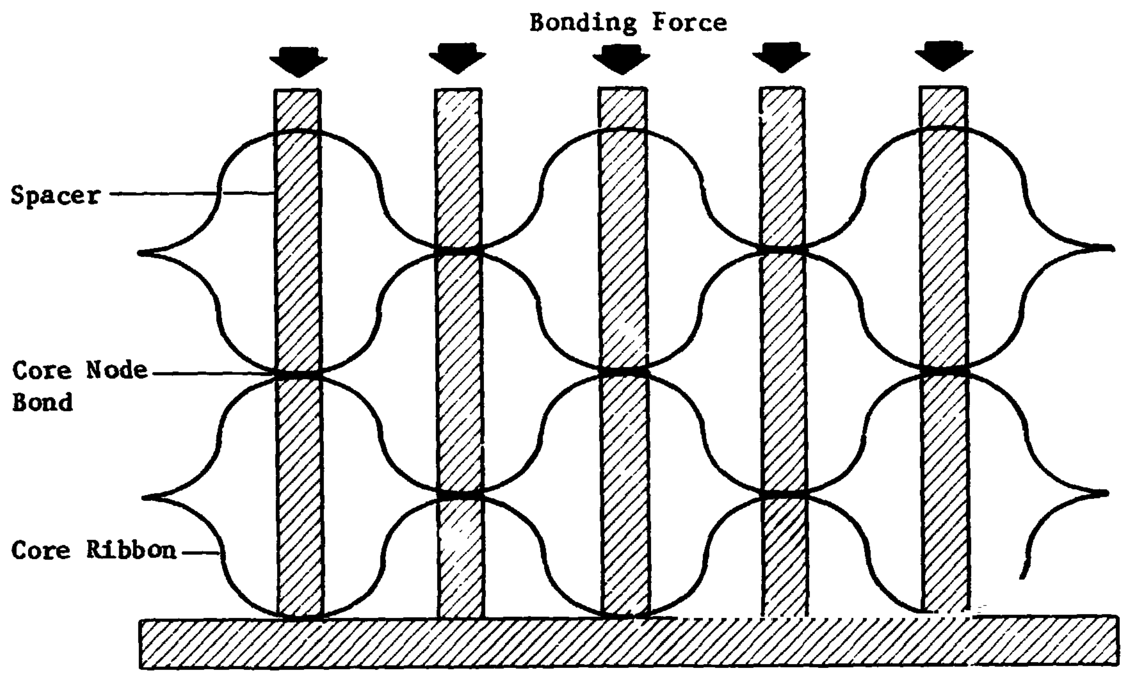
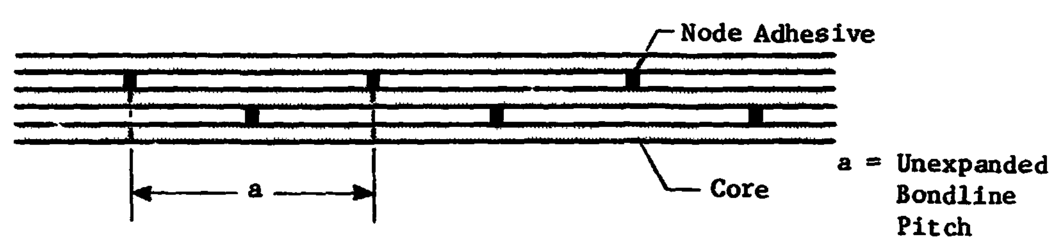
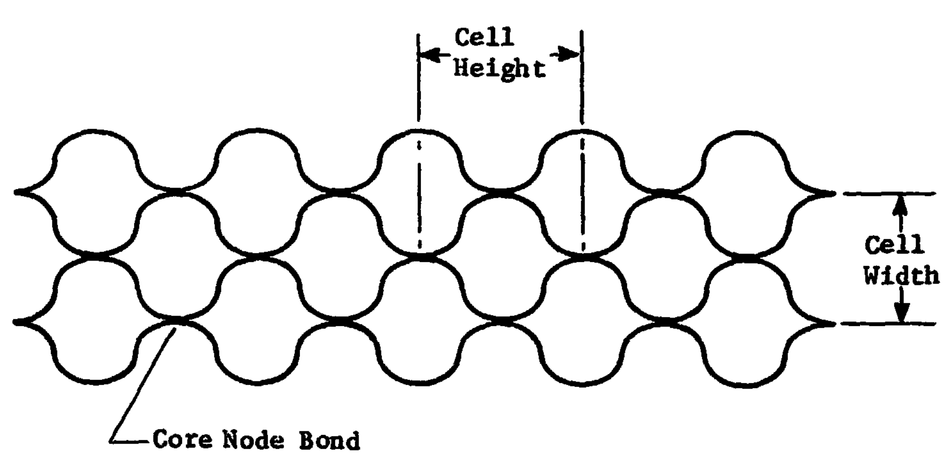


Fig. III-2 Core Bonding in Expanded Form



(a) Plan View of Unexpanded Honeycomb



(b) Plan View of Expanded Honeycomb

Fig. III-3 Plan Views of Honeycomb before and after Expansion

Problems were encountered in forming the rectilinear core with straight ribbons to a compound curvature (i.e., for dome cycle test specimens). In one approach to this problem, we fabricated the rectilinear core with a built-in compound curvature using precut Kapton ribbons to conform to tne shape of the dome when the core was expanded, and then bonded the ribbons in expanded form. Figure III-4 shows the templates used to cut the core ribbons prior to bonding, and Fig. III-5 shows a dome insulation specimen fabricated using this method. Although this technique was satisfactory for f ,ricating dome test specimens, it would not be practical for insulating a concentric tank.

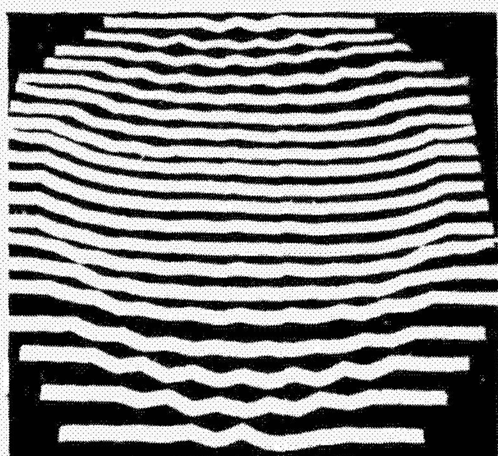


Fig. III-4 Dome Templates

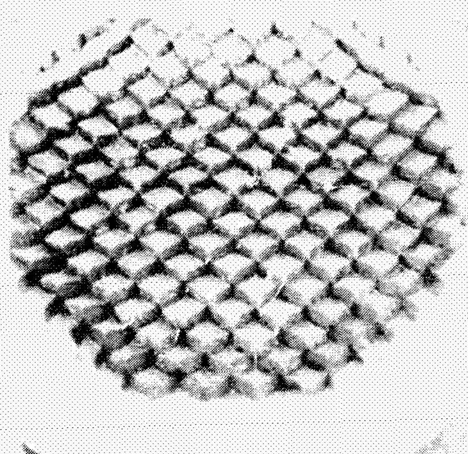


Fig. III-5 Core Bonded to Dome

The basic rectilinear core with straight ribbons was acceptable for insulating barrel sections of cylindrical tanks, provided that the installed curvature was not too great. Although an exact curvature limit has not yet been established, we have found that this core design can be used whenever the ratio of tank diameter to insulation thickness exceeds 60. For insulating cylindrical sections with smaller ratios, the rectilinear core can be formed with curved ribbons, as shown in Fig. III-6.* The following paragraphs discuss a similar solution for insulating sections with high compound curvature.

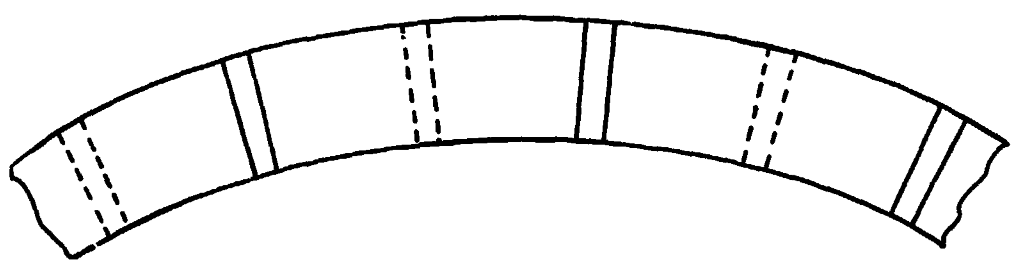


Fig. III-6 Flat-Pattern Fabrication of Rectilinear Core for a Small Cylindrical Tank

Panel Joint Development - The original concept considered for joining insulation panels during installation was a splicing operation that involved cutting the panels to the desired size and shape, installing them on the tank wall, and completing partial cells between the panels. This required bonding additional core ribbons (or splicing adjacent ribbons), installing filler material, and bonding, dimpling, and perforating the facesheet. Although only small splices were made using this concept, it was judged impractical for large-scale production.

---

*It is interesting to note that, when the insulation is expanded to follow a cylindrical contour, the ribbons follow an essentially straight path, parallel to the axis of the cylinder. The curvature of the ribbons in the flat pattern provides for the same expansion ratio (excess ribbon length factor) at the wall and facesheet sides of the core.

In examining alternative design concepts, we conducted further studies to improve a nested joint approach that had previously been developed for joining the insulation panels. In this basic concept, the panels are designed to nest together on all sides when installed, forming a narrow, serpentine gap between the panels. This gap is then filled using a grouting compound with a relatively low thermal conductivity.* A typical grout joint is shown in Fig. III-7.

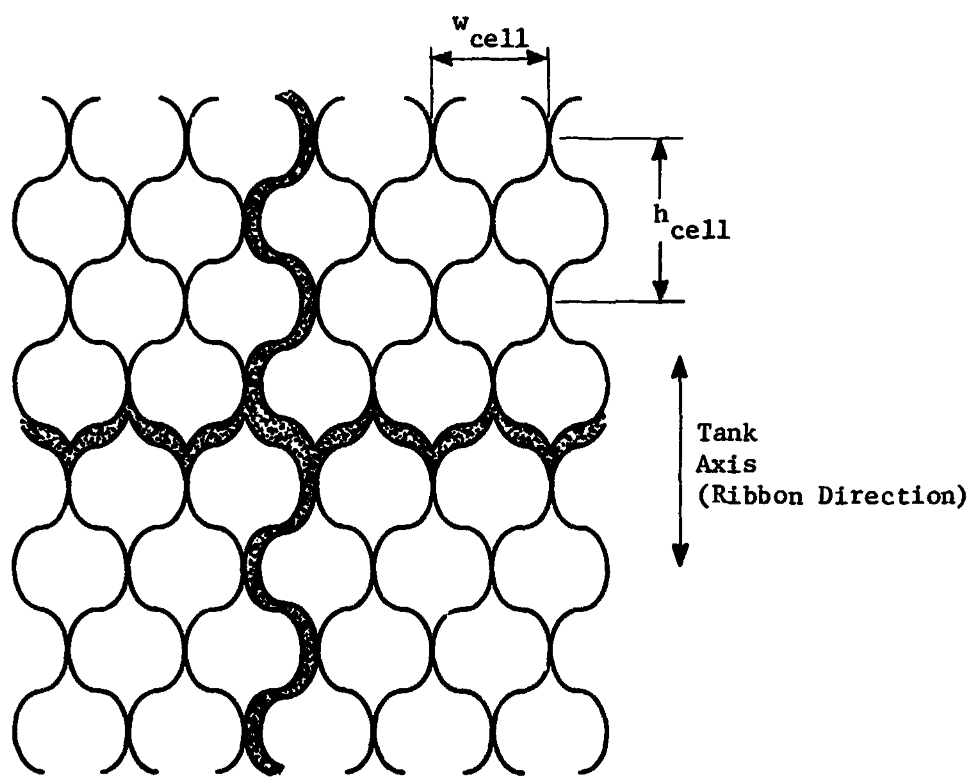


Fig. III-7 Nested Grout Joint Concept

---

*Despite the fact that the grout has a thermal conductivity several times higher than that of the insulation panel, the total cross section of the grout is on the order of 1% of the total area. Thus, there is no significant degradation in the performance of the overall system.

The curved grout joint can accommodate thermal contraction by changing shape, and consequently does not develop severe stresses. Using the grout joint method, the insulation panels must be designed to the shape and size of the particular tank to be insulated, but reasonable tolerances can be provided by using a thicker section of grout material. Each core panel is designed with an odd number of ribbons and an even number of core node bond lines. This results in panels whose length and width correspond to an odd number of "half" cells. In this context, a "half" cell refers to the total width of the panel in terms of the width of each cell, and not to incomplete or open cells. In other words, the panel's dimensions are equal to

$w = (n + \frac{1}{2})\, w_{cell};$

$h = (n + \frac{1}{2})\, h_{cell},$

where

n = number of cells in width or length of panel,

w = panel width,

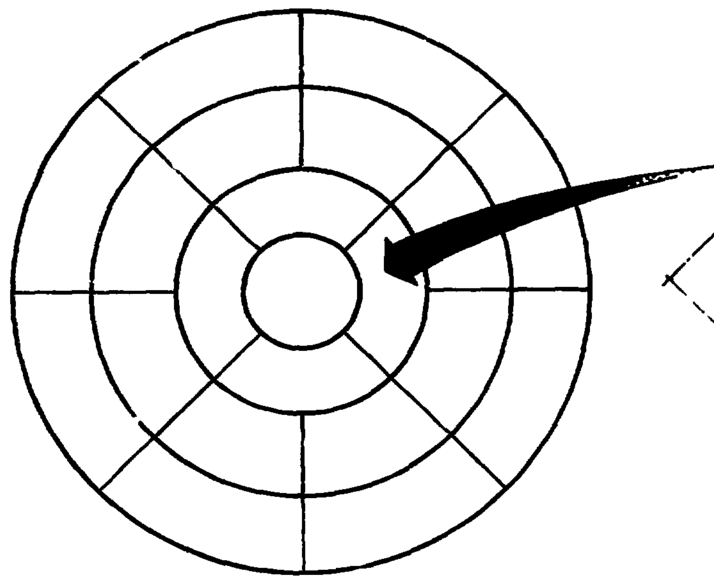
w_{cell} = cell width,

h = panel height,

h_{cell} = cell height.

Panel Design for Curved Domes - A special core design was developed to enable us to insulate an axisymmetric curved dome using the nested-grout-joint approach. For this application, the panels are designed in ring segments that are installed in concentric rings about the axis of the tank (Fig. III-8). All core ribbons radiate from the center of the dome. The number of ribbons doubles at discrete points as the distance from the centerline increases, thus increasing the number of cells and keeping cell dimensions within a fixed range.

The panels are designed with curved core ribbons to conform to the contour of the tank. Each panel nests with its neighbor on all sides, and the joints are grouted in the same manner described above (see Fig. III-9).



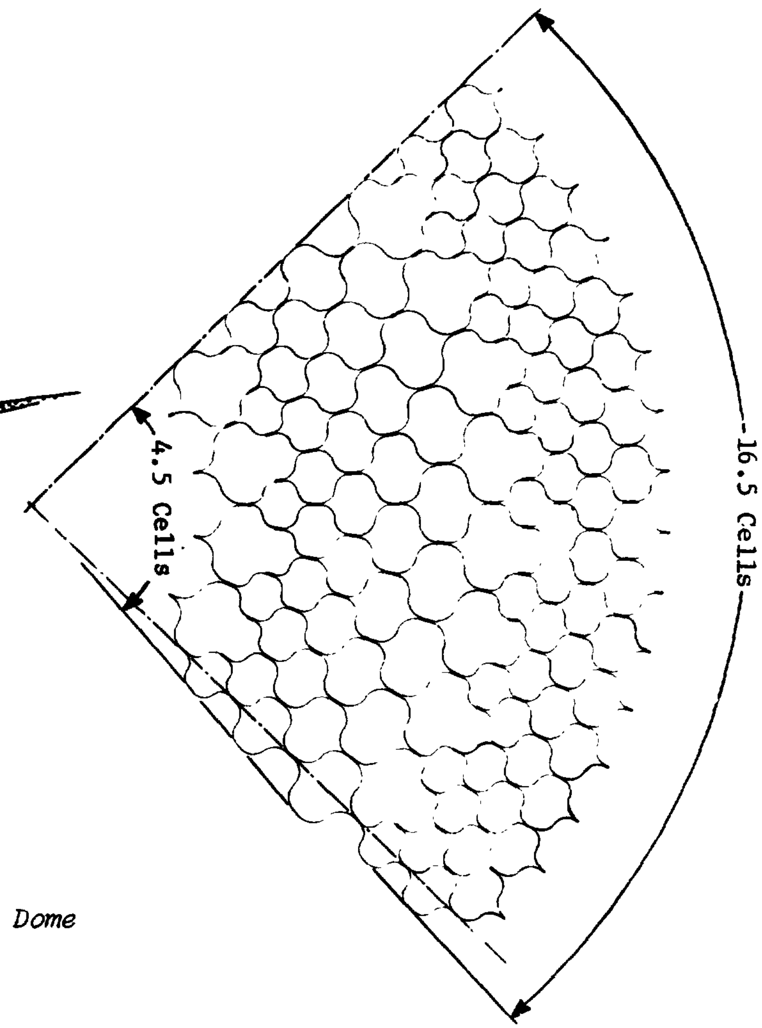


Fig. III-8 Arrangement of Panels in Tank Dome

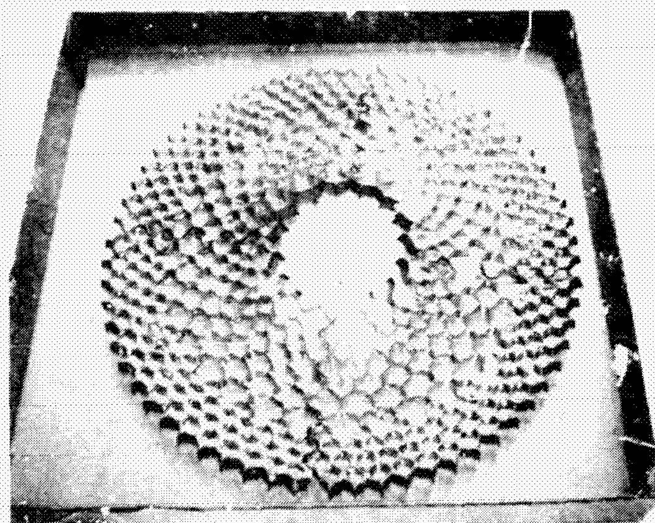


Fig. III-9 Four Nested Joint Panels Arranged to Form Complete Ring

For this application, the core is fabricated in a flat pattern with curved ribbons, and expanded to a compound curvature before the facesheet is bonded to it. The same approach is used to fabricate insulation for the transition between dome and barrel sections. In general, the core bond lines are perpendicular to the tank wall, but they may be tilted slightly to negotiate areas of high curvature, such as the dome/barrel transition in small tanks.

This method was used successfully in fabricating the aluminum and Inconel domes used in the dome cycle tests (see Chapter IV). The aluminum dome was insulated with two semicircular panels. The Inconel dome was insulated using four pie-shaped panels. (Previous test domes had been insulated with rectilinear core using curved ribbons.)

In designing insulation panels for a tank with an axisymmetric dome, the number of panels is selected as 2^n and the number of ribbons in each panel is selected as $2^n + 1$. This allows the

number of ring segment panels and ribbons to be reduced by one-half toward the center of the dome of a concentric tank, or doubled several times as their distance from the centerline increases, to keep the panels and cell size within a convenient size range.*

The method of designing the core ribbons to fit the curvature of the dome when the panel was expanded was based on the principles of descriptive geometry, but involved some trial and error. A more efficient and accurate method would be to use computer techniques and plotting, both to size and lay out the panels and to generate detailed panel designs. Such a computer-developed design was previously demonstrated for flat-bottomed tanks.

The nested grout joint concept and the radiating core design used for dome insulation were originated under a Corporation-funded commercial venture project. These concepts were further developed and refined on this contract effort. Details of fabrication methods and tooling concepts are discussed in Section C and Appendix B.

Under Contract NAS3-14384, *Internal Insulation System Development*, they have been extended to the design and fabrication of panels to insulate the entire inside surface of a 6-ft-diameter aluminum tank. Under that same contract, tooling and insulation panels were fabricated for tank barrel sections, domes, and the transition section between the barrel and dome. Although some improvements have been made during this tank demonstration effort, the basic design and fabrication concepts have proved successful.

In laying out the insulation for the 6-ft-diameter tank to be insulated under Contract NAS3-14384, we determined an optimum cell configuration for the cylindrical insulation panels. This optimum cell has a height (between bond lines) of 2.04 in. and an average width of 1.73 in. The bond line is 0.1875 in. wide and the pitch of the unexpanded bondline is 2.875 in.

These dimensions were chosen to provide an acceptable cell shape and to provide for eight panels, each 16 cells wide, extending around the circumference of the tank. On a larger tank (e.g., for the Space Shuttle, the cell size could be further optimized to minimize the number of core ribbons.

---

*Actually, the number of ribbons remains $2^n + 1$, where n decreases by 1 as ribbons are discontinued to increase cell size. This design is illustrated in Fig. III-8.

C. FABRICATION TECHNIQUES

Techniques for fabricating the internal insulation were evaluated using various material systems. These techniques are discussed in the following sections. A detailed process specification for fabrication is contained in Appendix ø.

Silicone adhesives were selected for the 350 and 650°F systems after considerable materials testing and fabrication experience. However, certain peculiarities were discovered in using silicone adhesives for this application. These are discussed at the end of this section under "Bonding Techniques."

1. Core Fabrication

Core design and fabrication methods for insulating a concentric tank were discussed in Section B. Before these concepts were developed, a number of techniques were used in fabricating test specimens to evaluate the effect of various materials systems.

The rectilinear core for the insulation test specimens was initially fabricated using Thermadite-17 as the core node adhesive. Due to the low viscosity and high bonding pressure required for this polyimide adhesive, the core was bonded in expanded form to prevent the bond lines from spreading. The adhesive was brushed onto 1-in.-wide Kapton strips at the desired spacing, and the strips were bonded in the three-dimensional frame shown in Fig. III-10. After the adhesive was cured, the core was expanded on a rake.

By using the RTV-156 silicone adhesive instead of the polyimide adhesive for core node bonding, it was possible to assemble the core ribbons in a flat pattern since high bonding pressures were not required. This provided precise control of bond line spacing and resulted in high fabrication efficiency.

For bonding the core with RTV-156 adhesive, the Kapton strips were soaked in water for a minimum of 12 hr and wiped dry within 30 minutes of applying the adhesive.

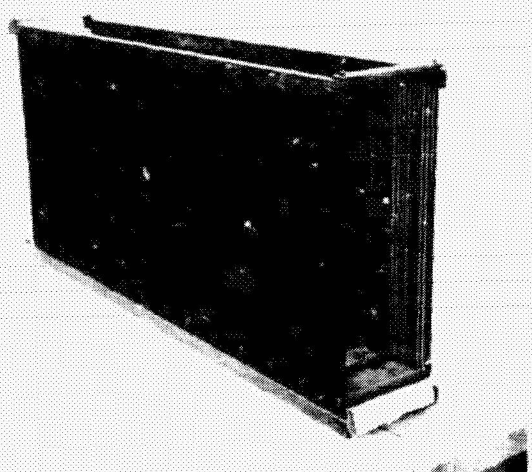


Fig. III-10 Stacking Tool for Bonding Core in Expanded Form

The adhesive was applied using a simple stencil made from 20-mil acrylic and a scraper blade. The stencil was installed in a printing fixture (Fig. III-11) that was equipped with a cover and a nitrogen purge system to extend the working time of the RTV-560 adhesive. To purge the printing fixture, nitrogen gas was fed through a tube attached to the inside of the printing frame. Small holes in the tube served as purge nozzles, minimizing the amount of moist air reaching the adhesive. This enabled us to work the adhesive for a minimum of 30 minutes.

The base of the printing frame was equipped with a punch to facilitate registration of the bond lines. After the ribbon was positioned in the frame, two 1/8-in. registration holes, spaced at exactly half the pitch of the bond line, were punched in each ribbon. Correct bond line registration was achieved by using a guide post to align alternate holes when stacking the ribbons. The stacking frame used to fabricate the rectilinear core is shown in Fig. III-12.

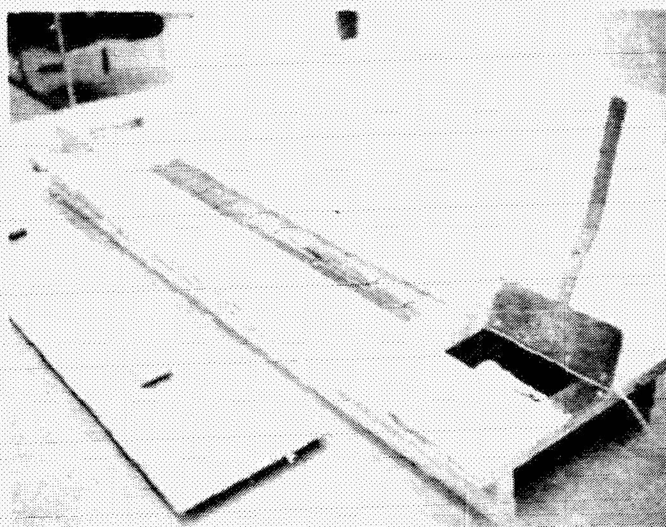


Fig. III-11 Core Printing Fixture

Fig. III-12 Core Stacking Fixture for Flat-Pattern Bonding

The ribbons were stacked against a rigid guide rail and forced into alignment by a flexible pad attached to a moveable frame. This provided close alignment of the ribbons on the facesheet side even when the width of the ribbons varied within permissible tolerances. A padded pressure bar was then placed on the completed stack to apply light bonding pressure.

The core assembly was cured in a humidity room for a minimum of 72 hr and postcured at 450°F for 4 hr. Section 8 discusses the development of bonding techniques and cure cycles for the RTV-156 core node adhesive. Figure III-13 shows a sample rectilinear core fabricated using this method.

The fabrication of dome insulation specimens (radiating core design) followed essentially the same procedure. Core ribbons were bonded in a flat pattern, trimmed to the proper outline, and expanded on a contoured assembly tool before bonding the facesheet. This fabrication method could probably be adapted to mass production by using die-cut Kapton ribbons. However, this type of manufacturing decision would have to be evaluated at a later date.

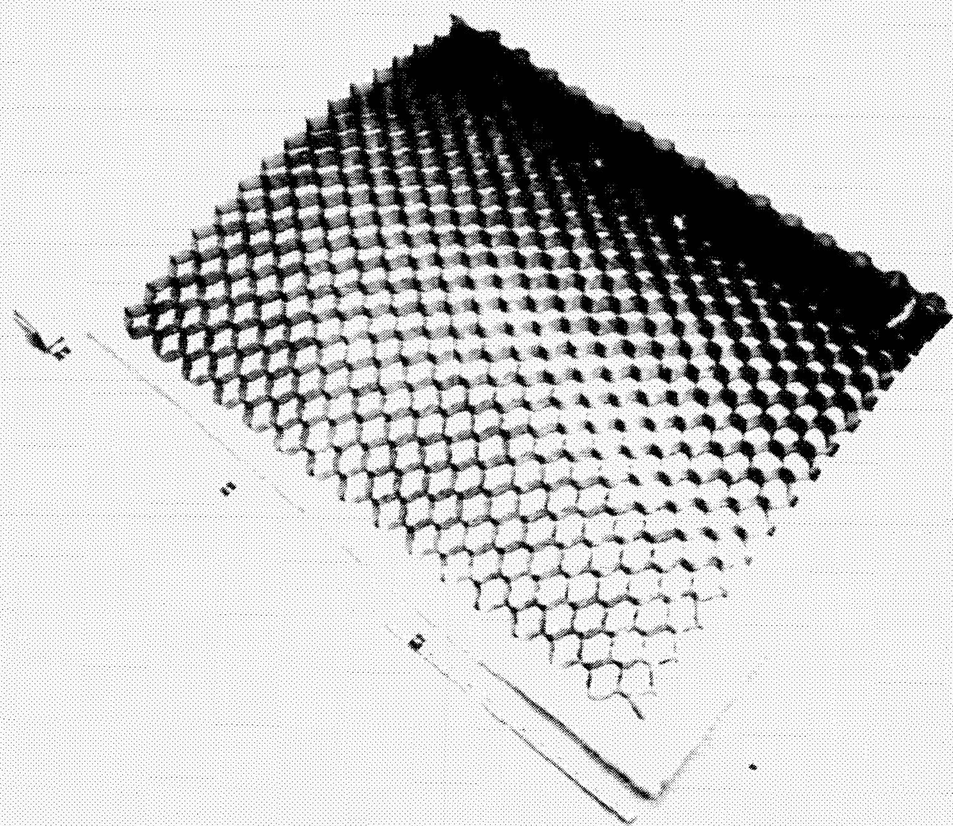


Fig. III-13 Example of Rectilinear Core for Cylindrical Tank Sections (Contract NAS3-14384)

2. Facesheet Bonding

The Kapton or Teflon facesheet was bonded to the expanded core on a contoured assembly tool with RTV-560 silicone adhesive. For test specimens fabricated with the polyimide adhesives (650-I system), a predimpled Kapton facesheet was used. A few of these specimens were bonded by applying adhesive to the facesheet in the pattern of the core. However, a more satisfactory method was to dip the core in the adhesive. For the selected materials system, the test specimens were fabricated by bonding an un-dimpled facesheet to the core, and then dimpling it.

The facesheet was cut to the approximate dimensions of the panel and silicone primer was applied on both sides. The core was then expanded on the assembly tool and sprayed with primer so that the ribbon surfaces were coated to a depth of at least 1/4 in. from the facesheet side of the core.

The assembly tool functioned both as a core expansion rake and a vacuum box for bonding and dimpling the facesheet. The assembly tool used to fabricate the cylindrical panels under Contract NAS3-14384 is shown in Fig. III-14. The tool for the aluminum dome panel is shown in Fig. III-15.

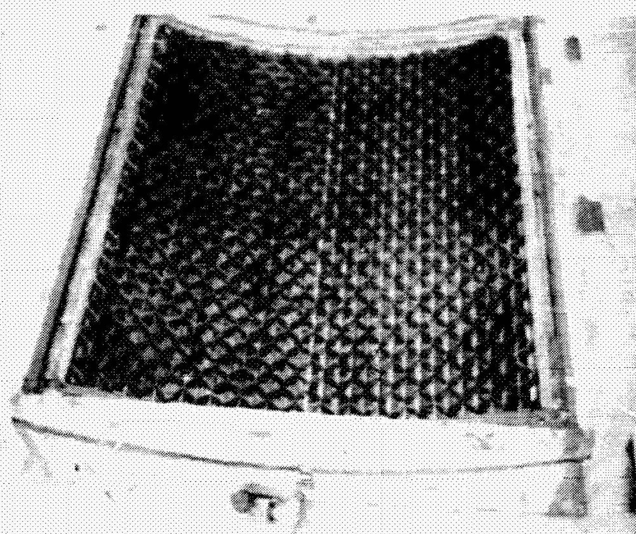


Fig. III-14 Assembly Tool for Cylindrical Panel with Core in Place (Contract NAS3-14384)

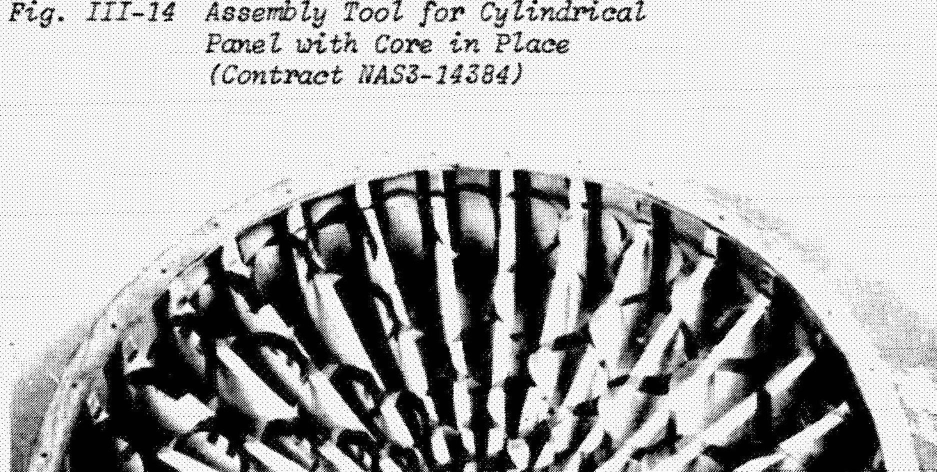


Fig. III-15 Assembly Tool with Core in Place for Aluminum Dome Panel

To bond the facesheet to the core, a uniform 1/16-in. layer of adhesive was first spread on a rubber or polyethylene transfer sheet and this sheet was assembled to the lid of the vacuum box. Next, the expanded core (intalled in the assembly tool) was inverted, and this was also assembled to the lid of the vacuum box. A slight vacuum was then used to draw the core against the coated transfer sheet. After approximately 3 minutes, the vacuum was released and the core was allowed to drip for 6 to 10 minutes. Originally, the core was blotted on a polyethelyene sheet to remove excess adhesive. However, this was unnecessary if sufficient time was allowed for the excess adhesive to drip off.

After the vacuum box/core assembly was removed from the lid, it was set aside in the inverted position. The adhesive transfer sheet was then removed and the facesheet was installed in its place. The vacuum box/core assembly was then reassembled to the lid and the facesheet was drawn against the core with vacuum (approximately 0.5 psid) for a minimum of 12 hr to cure the adhesive. This method ensures intimate contact between the facesheet and the core ribbons, which is essential to achieve consistently good bonding.

In fabricating some test specimens, we used a dead weight, rather than vacuum pressure to bond the facesheet.

3. Facesheet Dimpling

a. Teflon - The Teflon facesheet for the 350°F insulation system was intially dimpled by using vacuum to stress the material and then using a heat gun to apply a blast of controlled-temperature hot air to the material. Although this method produced fairly consistent results, it had two major limitations: the success was somewhat dependent on the skill of the operator, and the temperature was not constant throughout the cross section of the airstream.

We conducted several experiments to improve the quality of the dimpling operation and to minimize the influence of operator skill. As before, the insulation panel (with the core bonded to the facesheet) was installed in a vacuum box and a vacuum was applied to stress the facesheet. Next, an electric heater blanket was positioned over the facesheet and light pressure was applied to ensure contact with the facesheet. Finally, the temperature of the heater blanket was raised to 430°F and held for 1 to 2 minutes at a vacuum of 1.25 psid.

The heater blanket method provided consistent dimples of adequate depth. However, at 350°F the dimples almost completely disappeared due to shrinkage (apparently, the Teflon did not reach a sufficiently high temperature to eliminate the "memory" of the film). We found that we were unable to achieve the higher temperature required for permanent dimples using the heater blanket without creating hot spots that melted the film. Radiant heating was also unsatisfactory because of the high heat transmissivity of the Teflon.

Satisfactory dimples were finally achieved by returning to the hot-air blast method. Although this method has certain drawbacks (particularly the wide temperature variation across the airstream), adequate heat-set dimples were obtained using a hand-held electric heat gun.

In attempting to improve this method, we found that by reducing the differential pressure across the facesheet to 0.36 psi or less, dimples would only form at higher facesheet temperatures.

This discovery was significant since using a higher dimpling temperature, the dimples were not affected by being exposed to air at 350°F. After considerable practice, the operators were able to produce satisfactory dimples with only an occasional melt-through. The precise film temperature required for dimpling has not been determined because of the difficulty in measuring the actual temperature of the Teflon.

To obtain consistently satisfactory results, we decided to fabricate an improved dimpling fixture (Fig. III-16 and III-17). This fixture consisted of a 10-kw heater that discharges a narrow stream of hot air approximately 36 in. long. The heater was mounted on a swing arm assembly that could be moved across the insulation panel at a constant distance from the facesheet. Since the Teflon film has occasional weak spots, some melt-through still occurred in using this fixture even though no regular pattern of hot spots could be detected. Repairing the damaged cells was straightforward and was considered an acceptable inconvenience.

b. Kapton - Dimpling the Kapton facesheet for the 650°F insulation system was initially a problem because the 5-mil Kapton core buckled under a pressure of 3 psi without dimpling. Various methods of dimpling the Kapton facesheet were investigated. We first attempted to dimple the facesheet on a nominal 2-in. cell of Kapton core by freezing water to support the core, but these attempts proved unsuccessful. We then decided to try using a predimpled facesheet.

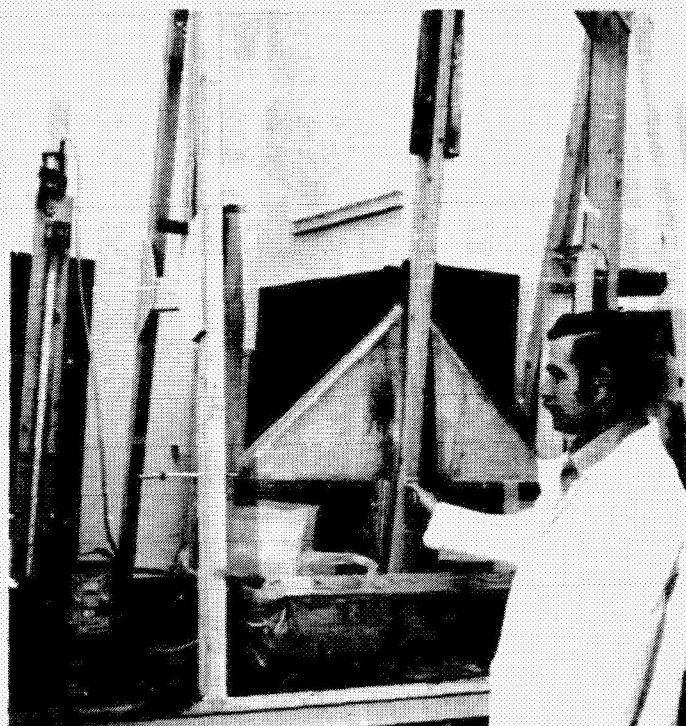


Fig. III-16 Dimpling Fixture

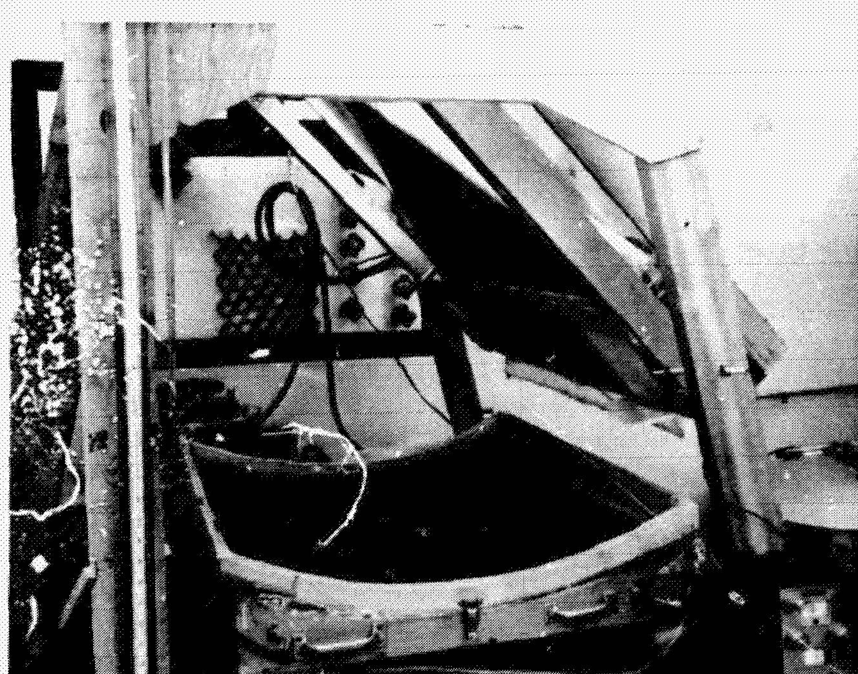


Fig. III-17 Insulation Panel in Dimpling Fixture (Contract NAS3-14384)

A metal plate was machined to the pattern of the core and attached to a vacuum box (see Fig. III-17). The Kapton facesheet was then predimpled on this tool in an autoclave at 500°F, using a differential pressure of 20 psi across the facesheet. This method was used in fabricating a number of test specimens.

Further experiments were conducted in an attempt to dimple the Kapton facesheet after bonding it to the core. The differential pressure across the Kapton film was increased by installing stiffeners in the core. These stiffeners were strips of thin shim stock (0.002-in. stainless) cut to the proper length to closely follow the cell wall when installed in pairs. This method allowed the pressure differential across the facesheet to be increased without collapsing the core. However, without increasing the dimpling temperature, no significant improvement was achieved.

A propane-fired heater was fabricated to achieve higher dimpling temperatures. Satisfactory dimples were obtained without having to increase the pressure differential above that which the core will support (approximately 1 to 2 psid).

Equally good dimpling results were obtained by modifying a standard electric heater gun to achieve a higher airstream temperature. An airstream temperature of 1000°F or more is required for dimpling the Kapton using this method. And although operator skill is required to prevent scorching, consistently good dimples can be obtained without damaging the Kapton facesheet.

4. <u>Facesheet Perforating and Trimming</u>

After the facesheet was dimpled, the excess facesheet material around the perimeter of the panels was trimmed close to the core, removing approximately half of the outside bond line. This manual operation required considerable care and patience. For mass production, a mechanized means for trimming the panels will be essential.

The facesheet was then perforated (one opening per cell) using a soldering iron with a 0.035-in.-diameter needle.

When the needle is heated sufficiently, the holes are partially melted through thus eliminating tears in the facesheet. By carefully controlling the temperature of the needle, and after some practice, satisfactory holes can be produced with a hand-held

soldering iron. However, if the iron is attached to a sliding mechanism, the temperature of the needle can be increased well above the melting point of the facesheet without producing elongated holes.

Figure III-18 shows a trimmed panel being perforated using this method. The perforating process is the same (except for the temperature required) for both Teflon and Kapton facesheets, and the perforating can be accomplished before or after the panels are trimmed.

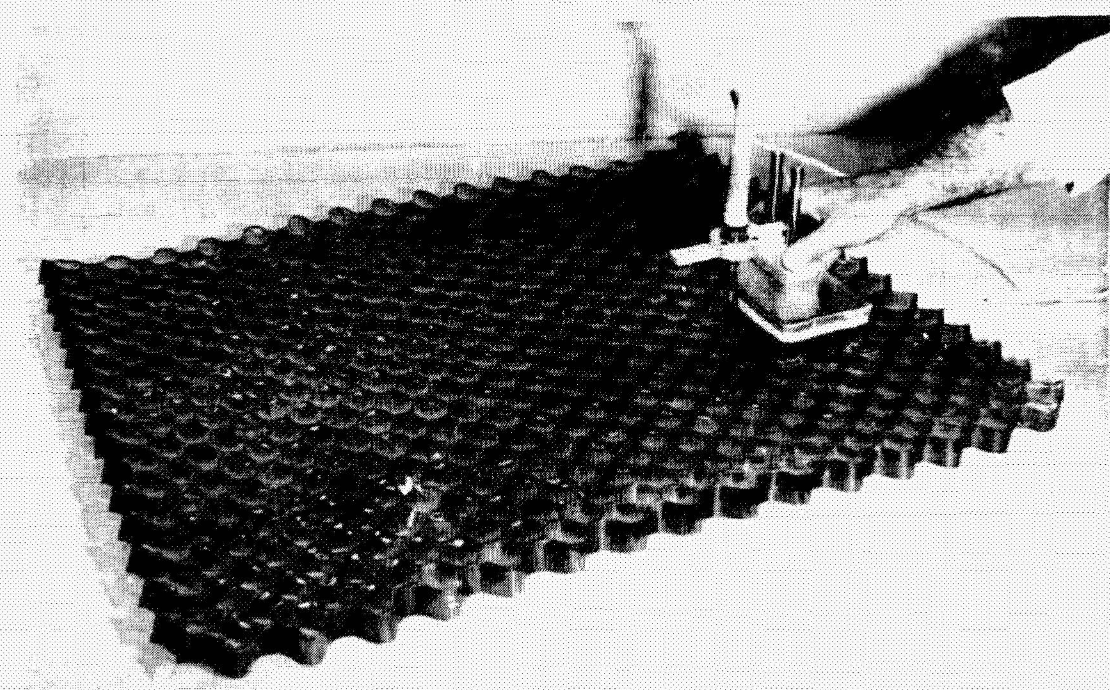


Fig. III-18 Trimmed Panel during Perforation (Contract NAS3-14384)

5. Installation of Filler Material

Several methods were evaluated for installing the fiberglass filler material in the insulation cells. We briefly considered grinding the filler material for bulk installation, but discarded this method because the ground material was too dense to be used in an insulation system with a reasonably low weight.

The most satisfactory method of installing the filler consisted of die-cutting plugs of 1/2-in.-thick PF 105 fiberglass batting to the desired shape (see Fig. III-19 and III-20). The filler plugs were cut 1% to 5% larger than the cells in length and width to provide excess material for compaction. Two 1/2-in.-thick plugs were used for each cell (for 1-in.-thick insulation). The filler material was then compacted to a uniform depth (1/32 to 1/16 in.) below the edge of the core ribbon. After installing the filler plugs, primer was sprayed on the tank wall side of the core to prime the core for installation and retain the filler material during handling.

For the polyimide adhesive specimens (650-I system), the filler material was sprayed with Thermadite 17.

6. Panel Installation

No complete tank was insulated as part of this program. However, the basic installation techniques that were developed are feasible and can be used in large-scale production. The following paragraphs describe our proposed method of installing the panels on a tank using silicone adhesives.

Panel installation requires the following steps:

1) Laying out the tank for proper positioning of the panels;
2) Cleaning and priming the interior of the tank;
3) Applying the adhesive;
4) Installing the panels in the proper location;
5) Holding the panels firmly against the tank wall until the adhesive is cured;
6) Filling joints between the panels with grout compound.

Fig. III-19 Pneumatic Punch for Fabricating Fiberglass Plugs

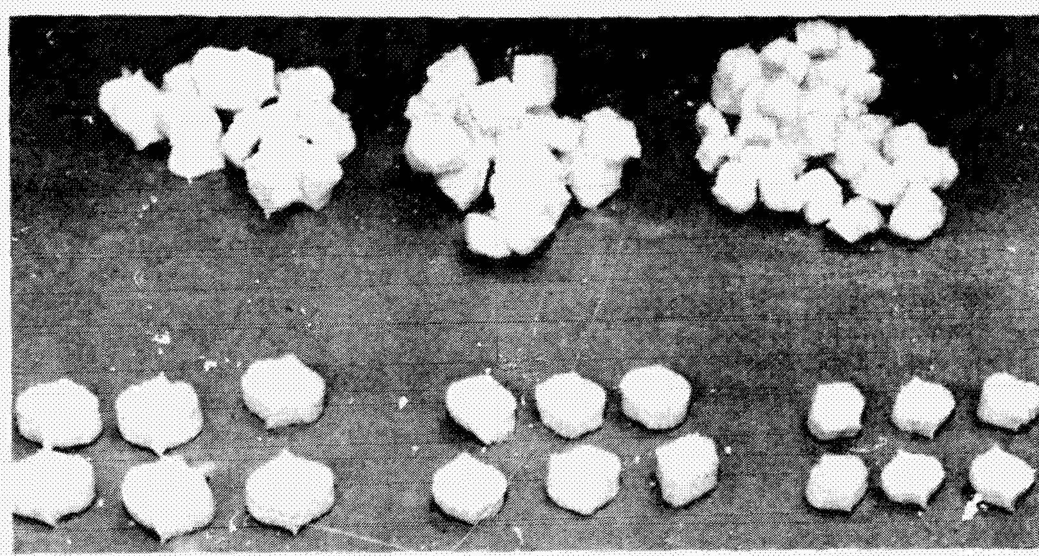


Fig. III-20 Typical Fiberglass Plugs

To minimize the weight of the insulation system and to optimize its thermal performance, the adhesive should be applied only to the extent needed to bond the insulation to the tank wall and prevent leakage between adjacent cells. One effective way to do this is to apply the adhesive with a stencil. This method is also more practical than dipping the core into the adhesive, because when the panels are filled with fiberglass batting, the loose fibers tend to contact the adhesive and pull parts of the filler out of the insulation. Furthermore, because of slight irregularities in the wall surface and the insulation, it is doubtful whether sufficient adhesive would be applied merely by dipping the core into the adhesive to prevent leaks between adjacent cells.

The stencils used to apply adhesive to the test specimens were made by bonding individual masks of 0.050-in. laminated flexible plastic to a 16-mesh polypropylene screen. The masks were cut to conform to the general shape of the core cells and were sufficiently undersized to produce fixed-width adhesive lines that followed the pattern of the core ribbons. The combined thickness of the mask and the screen controlled the thickness of the adhesive to approximately 1/16 in. Our experience with the test specimens demonstrated that a 1/16-in. layer of adhesive was sufficient to prevent cell leakage. The following paragraph describes our method for installing the insulation panels on the test specimens. This same method would be used in actual tank installation.

The stencil is positioned on the tank wall with the mask side nearest the wall, and the adhesive is applied using a rubber spreader. After applying the adhesive, the stencil is removed carefully to avoid smearing the adhesive. The core is then applied to the wall using adequate guide tooling to accurately position the panels so that they register with the applied adhesive. The guide tool and the stencil both use common index marks or locating buttons that may be temporarily or permanently bonded to the tank wall.

The core node points must be properly positioned while bonding the facesheet to provide close registration with the stenciled adhesive. Our approach was to design the panel assembly tool with spacer blades that extend between pairs of node bonds. Figure III-21 shows this concept being applied to fabricate panels for the 6-ft tank insulated under NAS3-14384.

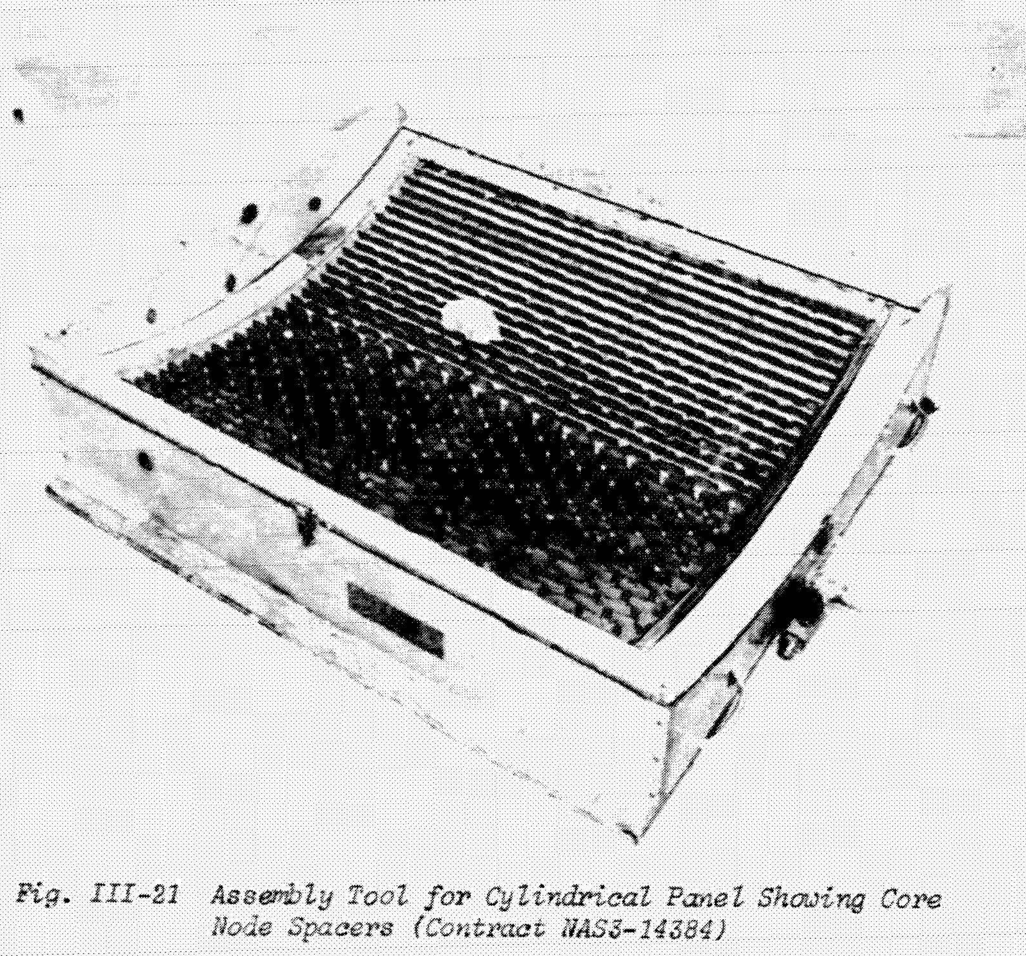


Fig. III-21 Assembly Tool for Cylindrical Panel Showing Core Node Spacers (Contract NAS3-14384)

Using this tooling method, the adhesive could be applied to the wall in as small a width as desired. Although we did not determine the minimum width of adhesive that was acceptable, our experience to date indicates that this width can be reduced to 1/4 in. or less for a 2x2-in. cell when production tooling is used.

The stencil application method was evaluated with the circular calorimeter and two of the aluminum dome specimens. The stencil and insulation panel for the calorimeter are shown in Fig. III-22. The panel is shown registered with the stencil in Fig. III-23. Figure III-24 shows the calorimeter plate after using the stencil to apply the adhesive.

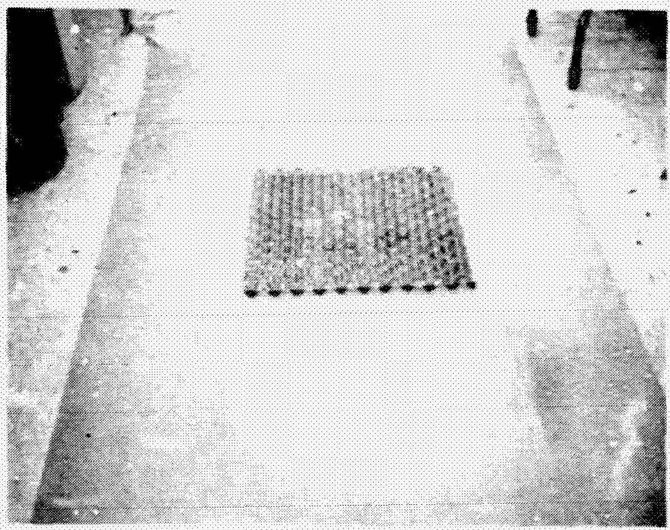


Fig. III-22 Stencil and Insulation Panel for Circular Calorimeter

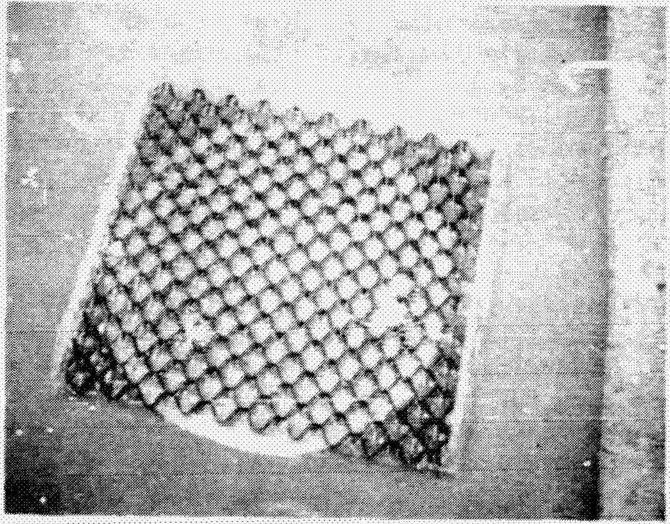


Fig. III-23 Insulation Panel Registered with Stencil

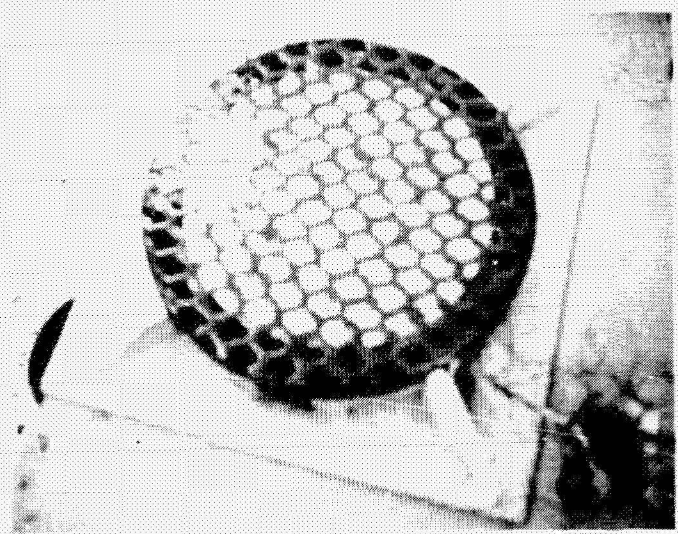


Fig. III-24 Adhesive Applied to Calorimeter Heater Assembly

For small, irregular areas, or when minimum weight is not a consideration, the adhesive can be spread uniformly over the tank wall with a notched rubber spatula. To permit the installation of panels on vertical and overhead walls, the viscosity of the adhesive can be increased by adding fumed silica (Cab-O-Sil, Grade M5). We found that adding approximately 1% of Cab-O-Sil gave RTV-560 adhesive enough viscosity for installing insulation panels on metal test articles. However, the proper amount to be used for actual tank installation will have to be determined by experience.

Several candidate grout formulations, all based on RTV-560 silicone adhesives, were examined. Compounds consisting of RTV-560 with 5 to 10% RTV-9811 curing agent and 1% to 2% Cab-O-Sil were found to be satisfactory for vertical joints. A grout formulation using 10% RTV-9811 and 2% Cab-O-Sil was used successfully in fabricating the thermal shock and dome specimens.

A Semco pneumatic applicator gun with a thin modified nozzle was used to apply the grout compound. (This application technique had been previously verified as feasible for commercial applications, using a compound of Crest 7343/7139 and prime milled cork.)

Figure III-25 shows a completed thermal shock specimen used to evaluate the nested grout joint.

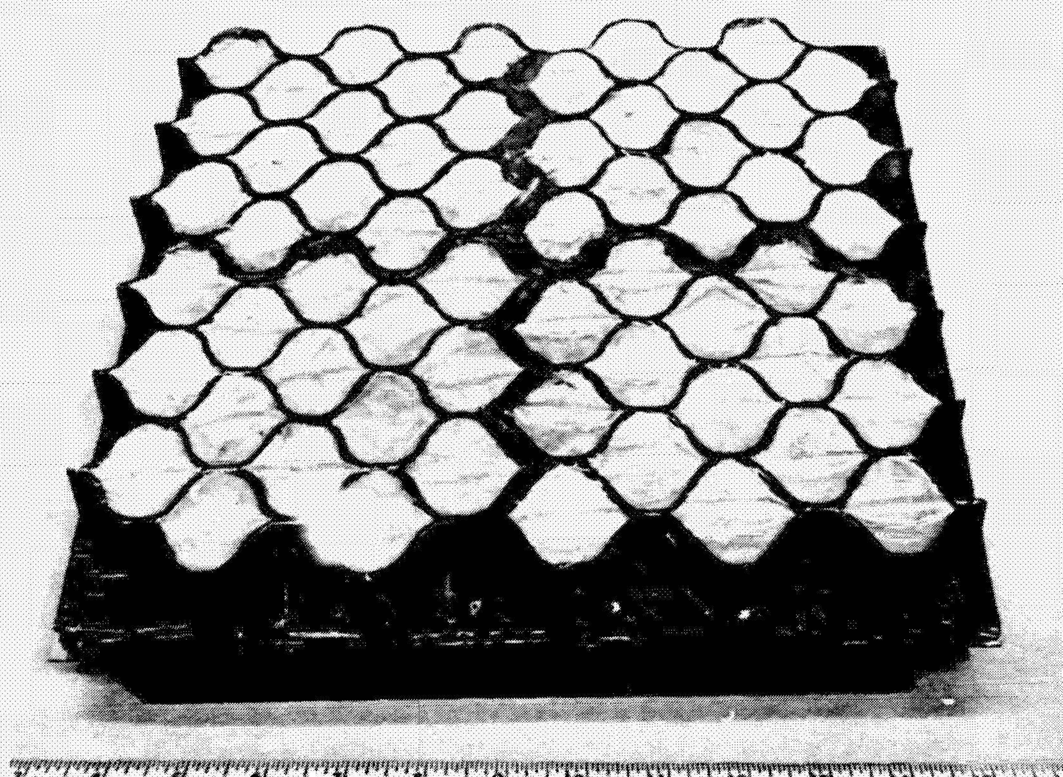


Fig. III-25 Thermal Shock Specimen Used to Evaluate Nested Grout Joint

To prevent leakage between cells, there mus. good contact between the insulation panels and the tank wall, and a minimum of bridging. This requires that an adequate, uniform bonding pressure be applied over the entire panel. This has been achieved satisfactorily by using a vacuum-bag technique, in which a pressure of 0.25 to 0.5 psid is applied to the panels to force them into conformance with the contour of the tank. Pressure pads can be used if the force is adequately distributed. The use of bags filled with lead shot to provide dead weight on the panels as they are being bonded to the metal test item has not proven totally satisfactory, and is not recommended.

We investigated several methods for cleaning the various tank wall materials. As a result of tests using both chemical and mechanical processes, the following procedure was selected for cleaning the tank wall.

1) Hand clean to remove paint, grease, and other obvious contaminants.

2) Vapor degrease with trichlorethylene.

3) Blast with new dry grit abrasive.

4) Blow off dust with clean dry air or nitrogen.

5) Verify cleanliness with water break or water drop spread test.

The size and type of grit are not believed critical. Aluminum oxide and silica, in grit sizes from 30 to 60, appear to give equally good results.

The air or nitrogen must be verified to be free of contaminants. This is accomplished by using chemically cleaned aluminum test plates. One of the plates is exposed to the airstream for 1 minute while the other, identically prepared plate is not exposed. The water-drop spread characteristics of the two are then compared, and no difference should be noted.

The above process is recommended for cleaning aluminum alloys and Inconel 718, and has been satisfactorily used with several test articles. In addition, it appears applicable for cleaning large tanks.

Chemical cleaning methods were also evaluated, but these did not significantly improve the quality of bonds to the aluminum alloys. For Inconel 718, we tried using a standard chemical cleaning procedure for stainless steel, which included degreasing, alkaline cleaning, nitric-hydrofluoric acid pickling, passivating, and deoxidizing, but this method did not produce satisfactory results during the water break test.

Priming must be accomplished as soon as possible after the tank is cleaned. The silicone primer (Dow Corning 1200) can be successfully applied by spraying, brushing, or wiping it on with a saturated cheesecloth. Spraying appears to be the method most applicable to large tanks. A sufficient amount should be applied so that it is visible after drying.

The installation procedures described above are being followed in insulating the 6-ft aluminum test tank under Contract NAS3-14384. For this tank, we plan to use the stencil only for applying the adhesive to the cylindrical panels. While curing the adhesive, all the panels will be held in place by a vacuum bag fitted to the interior of the entire tank.

7. Quality Control

As presently envisioned for boost-type hydrogen tanks, the capillary internal insulation system will have from 40 to 100 cells per square foot. Because of the large number of insulation cells involved, quality control procedures should minimize the need for a cell-by-cell inspection. Ideally, the quality of the insulation panels and their installation would be assured by process control and only spot inspections would be required.

During the development of design and tooling concepts and fabrication techniques, the tooling, equipment, and fabrication procedures were often inadequate to ensure consistent quality, and considerable rework and repair was necessary. For many of the test specimens, the initial inspection methods and criteria were not adequate to determine all of the defects, and satisfactory repair methods were unavailable. These defects were frequently due to incomplete bonding, excess adhesive, improper cell shape, and inadequate dimpling. As a result, we conducted an extensive program to develop tooling and methods for achieving consistently high quality panels. The development tools that were used to fabricate test specimens did not totally achieve this goal, though they did serve to verify the concepts. And since the installation methods that were chosen are based only on our limited experiences with the test specimens, they are subject to future verification and improvement.

The quality of the insulation panels was controlled primarily by visual inspections at six points in the fabrication sequence. These inspections were performed--

1) after printing, assembling, trimming, and postcuring the unexpanded core panels;

2) after priming the core panel and facesheet;

3) after bonding the facesheet to the core;

4) after dimpling the facesheet;

5) after perforating the facesheet and trimming the excess material;

6) after the filler plugs were installed.

A quality control document served as an inspection checklist, a record of acceptance, and an authorization to proceed to the next step. This "Panel Inspection Record" is presented in Appendix C, along with the detailed criteria against which the panels were inspected.

The above checks were largely visual inspections. However, when bonds were considered to be marginal, we used a leak test (see Fig. III-26) to verify that the seal between the cells was complete. This test was performed using a nitrogen pressure source and a rotometer-type flowmeter.

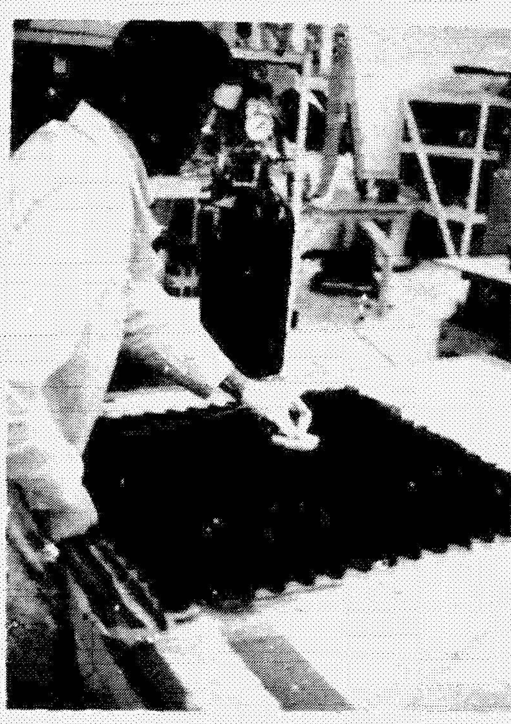


Fig. III-26 Leak Checking

To accomplish the test, the cells were pressurized from the open (wall) side using a flat, soft rubber seal pad connected to the flowmeter by a hose. The nitrogen pressure was regulated to 2 psig with the flow shut off at the seal pad. The pad was then pressed onto the open side of the cell and the flowrate was noted. Leak rates below 0.01 scf/hr can be detected using this method.

The same test was used to check for leaks after the panels had been installed. In this case, a smaller nozzle was pressed to the facesheet so as to cover one cell at a time, allowing the cell to be internally pressurized. Leaks large enough to be detected with the flowmeter were considered unacceptable.

In addition to leak-checking the cells, our postinstallation checks included visual inspections to verify the placement of the panels, to detect unacceptable distortion of the cells, and to verify absence of voids in the grout between the panels.

8. <u>Bonding Techniques</u>

We performed a number of tests to evaluate various primers and curing techniques for the silicone adhesives, and developed fabrication procedures that produced satisfactory results for both the 350°F and 650°F insulation systems.

a. Core Node Bonding: RTV-156 - The RTV-156 silicone adhesive proved to be an excellent material for core node bonding. However, certain pecularities were discovered in using this material with Kapton ribbons.

A number of the Kapton peel test specimens, which were initially fabricated using an adhesive thickness of 0.003 to 0.005 in., showed greatly reduced strength when tested at -320°F. The peel strength for these specimens generally ranged from 0.5 to 2 lb/in., compared with a minimum value of 5 lb/in. at room temperature. However, a few of the specimens exhibited higher peel strengths--of 8 lb/in. or more. In many instances the peel strength decreased after periods of several weeks or several months. Adhesives literature and discussions with General Electric personnel and other experts in the field did not provide ready explanations or solutions for these problems.

We discovered that the bonds were highly sensitive to moisture. During peel tests, certain aged specimens showed good bond strength, and failure occurred in the adhesive. However, if one breathed directly on the bond while a moderate peel force was being applied, the bond failed rapidly at one of the Kapton surfaces. In other specimens, debonding occurred when liquid water was applied to the bond. However, when the bonds were not caused to fail, even after they were thoroughly soaked, they regained their original strength when allowed to dry.

These results indicate that a highly soluble chemical bond is formed between the Kapton and the silicone adhesive under certain conditions. In general, when the bond loses strength in the presence of water at room temperature, it also loses strength at -320°F.

The moisture-sensitive bond, when it does occur, seems to depend on the thickness of the adhesive layer. No problem was encountered when the adhesive was thicker than 0.020 in.; in fact, the average peel strength of the bond was greatly increased. However, when the adhesive was thinner than 0.006 in., the core bonds ultimately lost strength to the point that they were damaged in handling, even without being exposed to moisture.

To eliminate this problem, we increased the thickness of the stencil from 0.010 to 0.020 in. to increase the thickness of the adhesive. When using a stencil to apply the adhesive, the practical maximum appears to be about 0.015 in., which is approximately the thickness achieved with the 0.020-in. stencil. Attempts to further increase the thickness of the adhesive resulted in spreading of the bond.

The problem of bond degradation when thin adhesive sections are used can also be eliminated by postcuring the core assembly. In an initial experiment with a bond thickness of less than 0.010 in., a postcure at 150°F for 4 hr appeared to eliminate the problem. However, this improvement proved temporary, and the bond strength decreased after several weeks. During subsequent experiments, we arrived at a postcure at 450°F for 4 hr, which has proven satisfactory.

Because of the delayed response, it was difficult to judge the adequacy of the 450° postcure treatment in improving the bond. One method that appears effective is to immerse a postcured specimen in boiling water for varying periods and then immediately measure the peel strength. By immersing the specimens in boiling water for periods as short as 30 minutes, we have been able to differentiate between cure cycles for specimens that had all previously survived a 16-hr water soak with no apparent loss of strength. Specimens cured at 450°F have survived the boiling water test and been aged for several months without losing bond strength. Consequently, this cure cycle was incorporated into the fabrication process.

After our initial experiments with RTV-156 core node bonds showed erratic peel test results, we experimented with a process wherein the Kapton ribbons were soaked in water for 12 hr and wiped dry within 30 minutes of applying the adhesive. This reduced curing time and seems to produce a higher and more consistent peel strength. The effect of soaking is related to the fact that the adhesive must absorb moisture to cure. Although soaking was not recommended by the adhesive manufacturer, our investigations verified that it had no relation to any of the bonding problems (i.e., the moisture-sensitive bond) discussed above, and it was incorporated into the fabrication procedure.

b. Facesheet Bonding: RTV-560 - To evaluate the RTV-560 adhesive for facesheet bonding, we fabricated a series of tubular specimens, approximately 2 in. in diameter, using either Kapton or Nomex for the cylinder, 2-mil Teflon for the facesheet, and General Electric SS 4004 and SS 4155 primers. The completed tube specimens were evaluated by internally pressurizing the tube. Figures III-27 and III-28 show typical specimens before and after the test.

Bond failures were experienced in some of these specimens. This problem was later recognized as being due to inadequate curing of the silicone primer. The specimens which were defective due to inadequately cured primer were detected readily, since the facesheets were debonded with only a negligible pull force. Except in these defective specimens, no adhesive failures occurred.

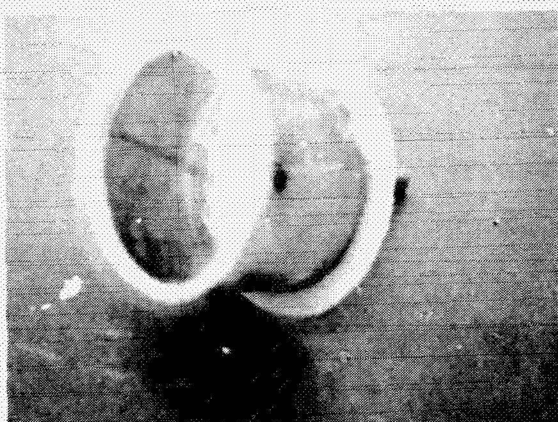


Fig. III-27 Facesheet Bond Test Specimen Before Test

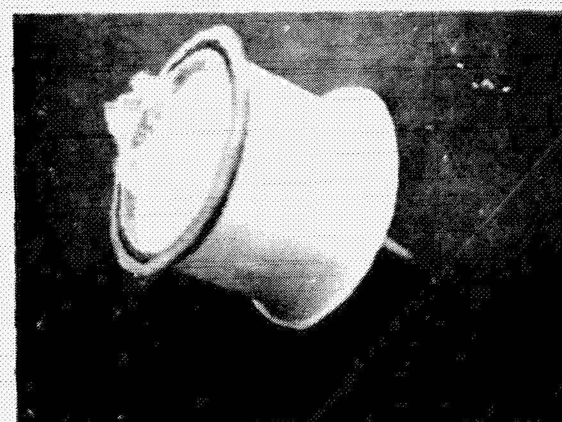


Fig. III-28 Facesheet Bond Test Specimen After Facesheet Rupture

At room temperature, the Teflon failed at approximately 10 psi; at the temperature of liquid nitrogen no failure occurred at 15 psi; and at 350°F, small pinholes developed in the Teflon at approximately 2 psi. The pi:holes were probably caused by hot spots well above 350°F.

c. Core-to-Metal Bonding: RTV-560 - A series of tension tests were conducted to evaluate the strength of the RTV-560 core-to-metal bond at elevated temperature. The test specimens were fabricated by bonding a single Kapton cell between two 2024-T6 aluminum blocks, as shown in Fig. III-29. The aluminum was cleaned by solvent degreasing, followed by mechanical cleaning with pumice and wet Scotch-Brite pads. Cleanliness was verified after rinsing the blocks by subjecting them to a water-break test. General Electric 4004 primer was then applied and allowed to air-dry.

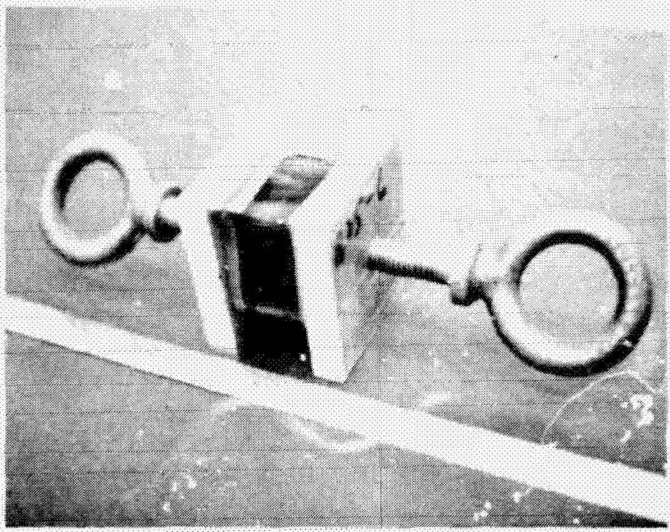


Fig. III-29 Bond Tension Test Specimen

The RTV-560 adhesive was applied by dipping the Kapton core cells to a depth of approximately 1/8 in. and allowing the excess to drip off. Two different curing agents were used: dibutyl tin dilaurate (Thermolite 12) at approximately 0.5%; and a tin soap curing agent, General Electric RTV-9811, at approximately 10%. Six of these specimens were tested in tension at 350°F and six were tested at room temperature. The results of the test were as follows:

Sample	Curing Agent	Tensile Load at Failure, lb	Test Temperature
1	Dibutyl tin dilaurate	36	350°F
2	Dibutyl tin dilaurate	43	350°F
3	Dibutyl tin dilaurate	39	350°F
4	RTV-9811 (Tin Soap)	38	350°F
5	RTV-9811 (Tin Soap)	38.6	350°F
6	RTV-9811 (Tin Soap)	40	350°F
7	Dibutyl tin dilaurate	61.0	70°F
8	Dibutyl tin dilaurate	42.5	70°F
9	Dibutyl tin dilaurate	62.8	70°F
10	RTV-9811 (Tin Soap)	52.0	70°F
11	RTV-9811 (Tin Soap)	56.2	70°F
12	RTV-9811 (Tin Soap)	52.4	70°F

The length of the bond for these specimens was 6 3/4 in. All failures occurred within the adhesive. Therefore, the minimum bond tension strength that was obtained was 5.3 lb per inch of ribbon at 350°F and 6.3 lb per inch at room temperature.

The RTV-9811 was used with the RTV-560 for core-to-metal bonding. RTV-9811 is a thick-section curing agent that is advertised to be independent of ambient humidity, and therefore does not require exposure to air. Although the bonding of prefabricated panels to the tank wall does not require thick sections of RTV-560 (in excess of 1/16 in.) the adhesive has only limited exposure to ambient air because of the enclosed location of the bond. Also, the ambient humidity may be inherently low or be controlled to a low level for other reasons.

Our experience with the RTV-9811 curing agent indicated that the cure time for thin sections with large surface area was greatly increased by a low relative humidity environment (less than 15%).

For bonding the insulation to the tank wall, we mixed the RTV-560 and the RTV-9811 curing agent with 1% by weight of Cab-O-Sil to give the adhesive the proper viscosity. This combination was used in preparing several test specimens, including those for the aluminum dome, the Inconel dome, and the circular calorimeter. Since the initial speciments which contained Cab-O-Sil required very long cure times, we conducted a series of tests with combinations of curing agents to find ways to speed up the curing process.

The combination of curing agents we selected for installing the insulation was 5% RTV-9811 with 0.25% dibutyl tin dilaurate. This reduced the curing time to an acceptable value, and the adhesive achieved a satisfactory strength in 16 hr. This same combination was used to formulate the grout compound.

d. Primer Selection for RTV-560 - Three primers were evaluated for use with the RTV-560 silicone adhesive:

1) General Electric SS 4004;

2) General Electric SS 4155;

3) Dow Corning DC-1200.

In specimens prepared using each of the three primers, bond failures occurred within the adhesive, rather than at the primed surface.

Since the SS 4155 primer forms a chalky layer that can be wiped off during handling, we eliminated it from further consideration.

Both the SS 4004 and the DC-1200 primers were acceptable for core-to-facesheet bonding (with both Teflon and Kapton facesheets) and for core-to-metal bonding. Initially, we believed that the SS 4004 primer offered advantages over the DC-1200 primer. Later experience indicated that both were equally acceptable, and that the differenc[illegible] noted were due to variations in our application and primer [illegible]niques. Primarily because of greater experience a[illegible]cts with the DC-1200 primer, it was selected for both t[illegible]C and 650°F systems. Using this primer, we have found that [illegible] surfaces can be stored for up to 10 weeks without impa[illegible] the effect[illegible]eness of the primer.

After the RTV-560 silicone adhesive had performed satisfactorily in numerous te[illegible]s, several unexplained bonding failures occurred. In a high percentage of cases, the adhesive totally failed to bond to Kapton and Teflon, even though there had been no apparent change in the materials or bonding procedures. Intensive investigation revealed that the failures primarily resulted from inadequate curing of the silicone primers due to the low humidity.

Although vendor literature, Martin Marietta procedures for the use of silicone adhesives, and local experts all recommended increased curing times for low relative humidity, these recommendations proved inadequate for the below-10% relative humidity conditions that we encountered.

To solve this problem, a 20-ft by 20-ft enclosure was built in the work area and equipped with humidifiers (see Fig. III-30). This humidity room provided a relative humidity of 40 to 60% at all times except weekends, when the humidifiers were not operating. All primer curing and bonding operations were subsequently performed in the humidity room.

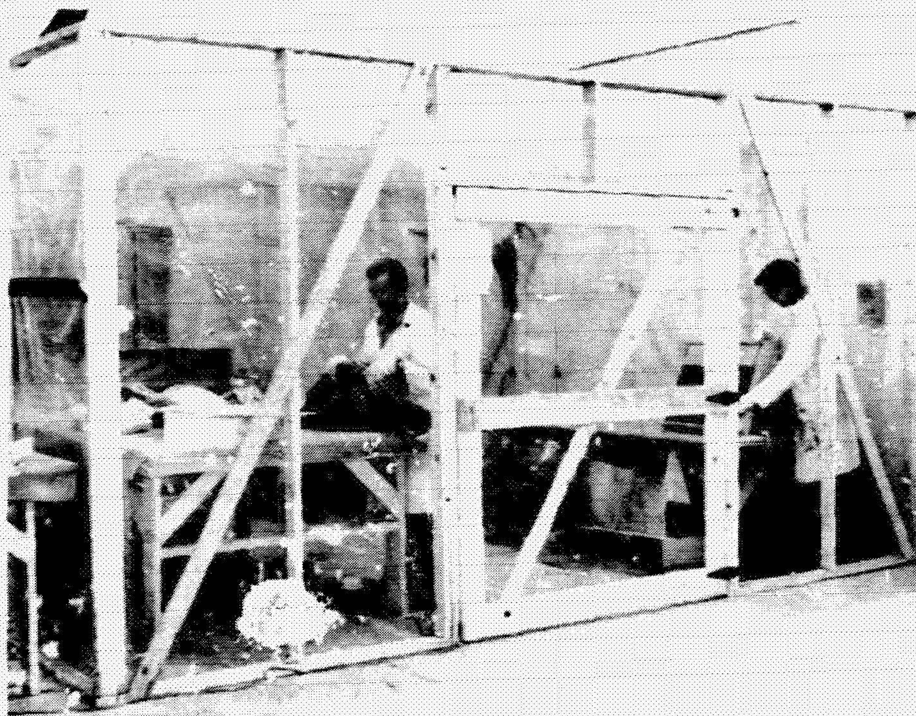


Fig. III-30 Controlled Humidity Room

Although hydrolyzation of the primers (either SS 4004 or DC-1200) was necessary, they could have been contaminated by premature exposure to a humid environment. Consequently, we applied the primers outside the humidity room and moved the specimens to the humidity room only after the solvents had completely evaporated. Since the cure time for the adhesive also depends on the humidity, a more consistent bonding schedule could be maintained with this procedure.

After being primed, the core panels and facesheets were cured in the humidity room for a minimum of 3 hr.

In addition to curing the primer, bonding it to the Teflon facesheet and the Kapton core also appeared to depend on the amount of primer applied. Although in some instances the minimum amount of primer that could be applied by wiping it on with a saturated cheesecloth pad was acceptable, we found that using several times that amount gave more consistent results with Teflon. The additional thickness of primer also provided a visible indication that the primer had been applied to the entire surface, whereas application of the minimum amount was difficult or impossible to detect visually.

If enough primer is used, it may be applied either by wiping with cheesecloth, brushing, or spraying.

e. Polyimide Adhesives - For the 650-I insulation system (polyimide adhesives), voids in the bondline were a recurring problem. As a result, a number of tests were performed to evaluate the outgassing characteristics of the BR-34 and Thermadite-17 polyimide adhesives. The test specimens were made of 5-mil Kapton core and a 1-mil Kapton facesheet.

For both adhesives, using a slower temperature rise reduced the incidence of trapped bubbles; and the elimination of excess adhesive was also effective in controlling this problem. A significant improvement resulted from using a dry nitrogen environment in the curing oven.

Bond line voids with BR-34 adhesive were most successfully eliminated by thinning the adhesive by 30 parts per 100 with standard BR-34 thinner supplied by the vendor. The core was dipped in adhesive to a depth of 1/16-in., then blotted on a Mylar sheet for 5 to 10 sec, and finally placed on the Kapton facesheet, which was supported on a soft pad.

Several cure cycles were used to achieve void-free lines. One of the more successful cure cycles was as follows:

Air-Dry	1 hr minimum
90°F	1 hr
195°F	4 hr
230°F	4 hr
300°F	3/4 hr
350°F	2 hr

The bond was postcured at a later time, in accordance with the manufacturer's recommendations. Figures III-31 and III-32 show an insulation test panel fabricated using this procedure. Although the polyimide bonds achieved with this process were satisfactory, the silicone adhesives proved to be more practical for fabricating the 650°F insulation system.

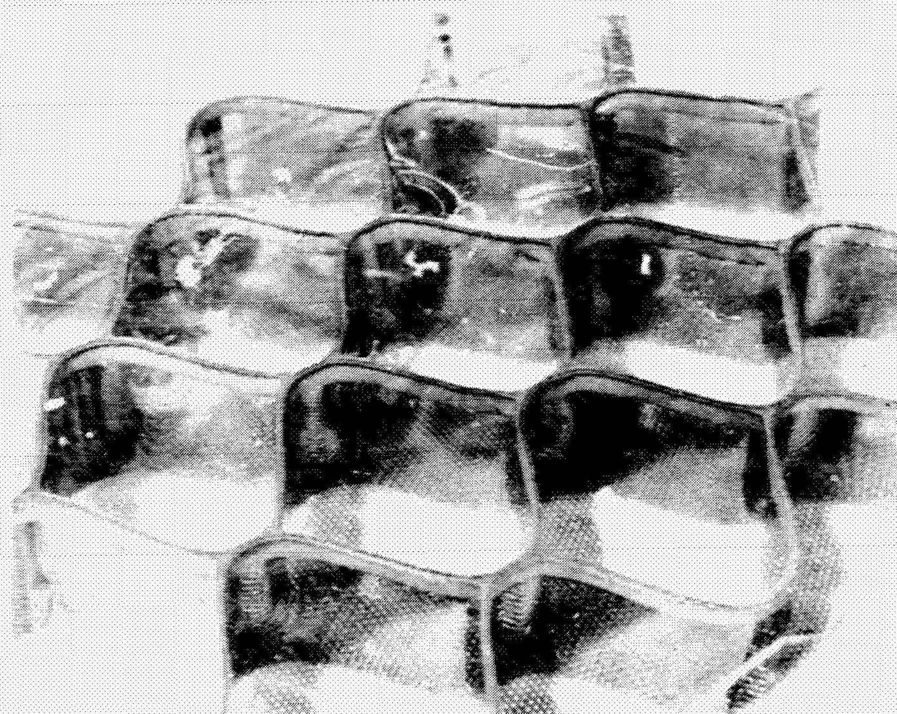


Fig. III-31 Polyimide Bonding Specimen - Facesheet Side

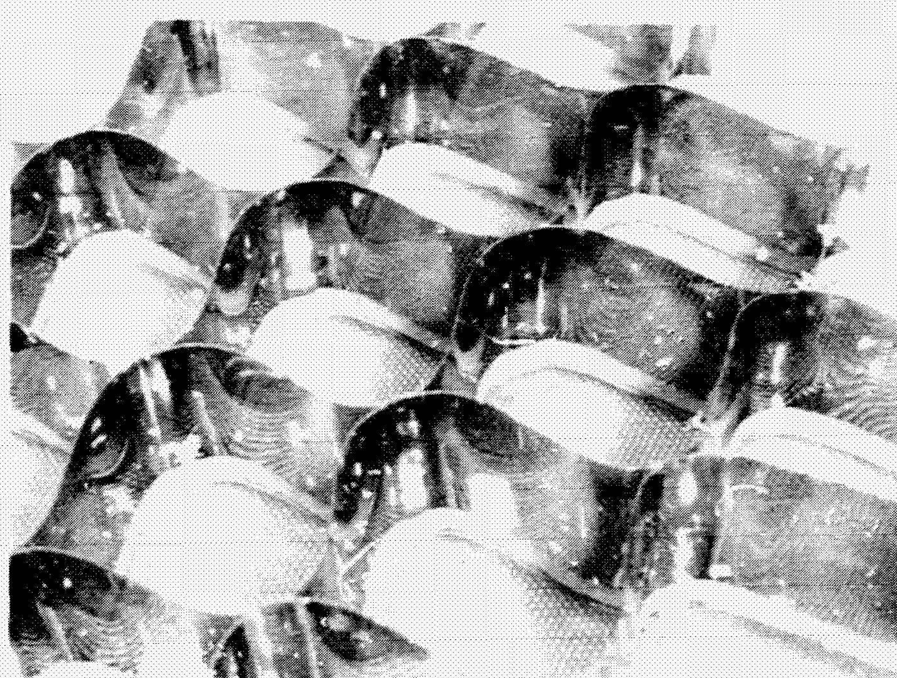


Fig. III-32 Polyimide Bonding Specimen - Tank Wall Side

D. REPAIR TECHNIQUES

In fabricating insulation panels and test specimens, we developed repair procedures to correct for several types of damage and defects that were encountered during this program. These are described in the following paragraphs, and apply generally to the Kapton, Teflon, and silicone adhesive materials.

The most common problem we encountered was one of skips in the facesheet-to-core bond, or very thin spots in this bond, which appear to be potential leak points. These are readily repaired by manually applying a thin bead of adhesive over the defect area, generally on both sides of the ribbon. This can be accomplished by using a narrow spatula or a hypodermic syringe with a 16-gage needle, filled with RTV-560 adhesive diluted approximately 5 to 10% with General Electric RTV-910 thinner. In either case, care must be taken to prevent excessive adhesive from building up and/or prevent spreading the adhesive over a larger area of the facesheet than is desirable.

In some instances, we found that cells on the perimeter of the panel were distorted because of improper fitting of the core to the assembly rake or because of inaccuracies in the tooling. These cells have been successfully repaired using the following procedure. The facesheet was first debonded from the core over the distorted area by failing the adhesive. A template of the proper size and shape was then inserted into the cell to hold the ribbon to the proper contour, and adhesive was applied on the outside of the cell at the facesheet-core intersection using a spatula or syringe. After this first adhesive cured, the template was removed and the bond was completed on the inside intersection of the core and facesheet. An extraneous line of adhesive will remain on the facesheet after this repair is completed. However, no resulting problem in subsequently dimpling the facesheet has been experienced. Figures III-33 and III-34 show a distorted cell before and after being repaired.

In a very few cases, tears occurred in the Kapton core ribbon, beginning from the open or wall side of the prefabricated panel. These tears have been repaired by bonding a small Kapton patch over the tear using RTV-156 adhesive.

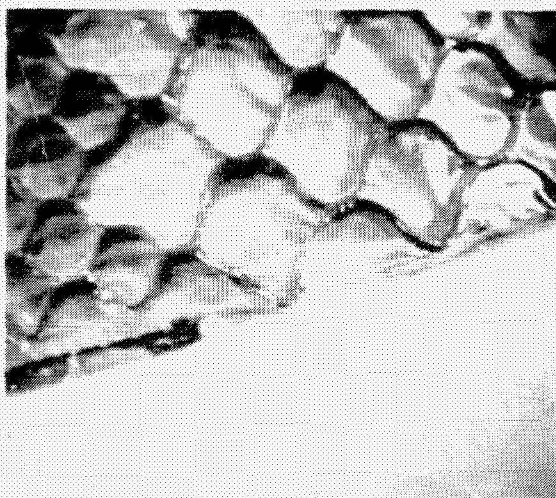


Fig. III-33 Distorted Cell on Facesheet Side before Being Repaired

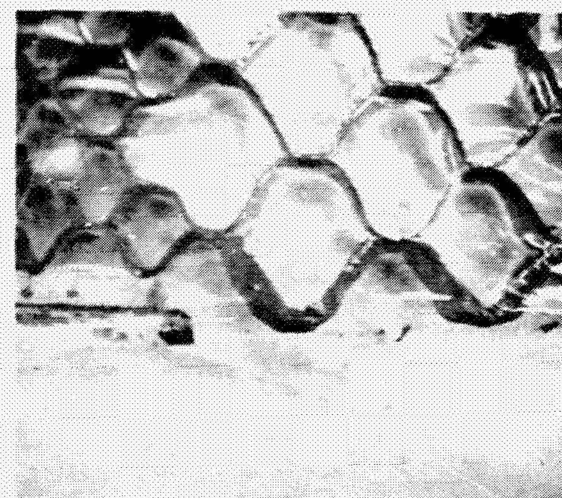


Fig. III-34 Distorted Cell on Facesheet Side after Being Repaired

In some instances the Teflon melted through in isolated cells during dimpling. In such instances, the facesheet was trimmed out for these cells using a sharp knife. A predimpled Teflon patch was then bonded directly over the remaining bond around the perimeter of the cell using RTV-156 adhesive. Figure III-35 shows a specimen with a facesheet patch.

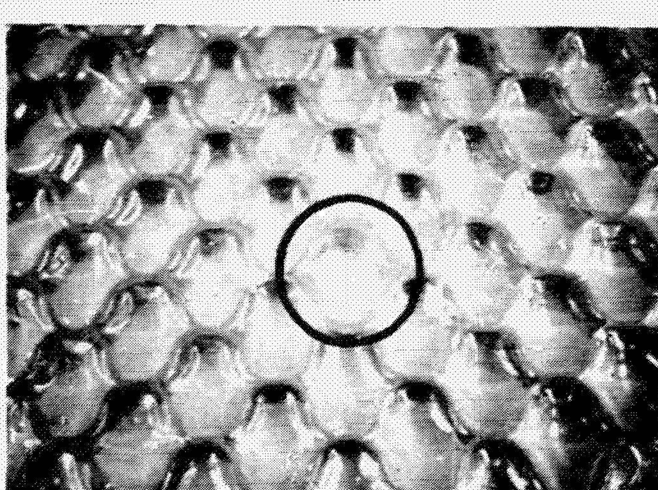


Fig. III-35 Specimen with Facesheet Patch

These patches can be cut from facesheets that had been removed from rejected panels after the dimpling operation was completed. No difficulty was experienced because of the RTV-560 that remained on the Teflon.

Small holes in the facesheet can be repaired by bonding a small Teflon patch over them, using either RTV-156 or RTV-560. This method should be limited to cases where the patched area is a very small part of the cell, and where the patch will not prevent satisfactory dimpling.

In trimming away the excess facesheet around the perimeter of the cell, the facesheet and adhesive will occasionally be completely removed up to the outer edge of the Kapton ribbon in small areas. This does not in itself affect the integrity of the cell, since the bond will be complete on the opposite side of the ribbon. Such overtrimmed spots are considered potential leak points, however, and are repaired by applying a thin layer of RTV-560 adhesive so that it overlaps the Kapton-Teflon intersection.

The above repairs apply to insulation panels that have not yet been installed on a tank wall. In general, they also can be used to repair damage that may occur after installation. For damage to large areas, however, our recommended method is to completely remove the damaged area, carefully trimming the facesheet to the boundary of the remaining cells. A special panel must then be fabricated. This panel will be slightly undersized, but can be bonded in place and grouted in the standard manner.

IV. INSULATION SYSTEM EVALUATION

To evaluate materials for the 350°F and 650°F insulation systems, insulation specimens were fabricated with the materials systems discussed in Chapter II and subjected to thermal shock tests, thermal conductivity tests, and dome cycling tests. An insulation thickness of 1 in. was chosen for all specimens as being typical for a reusable booster hydrogen tank. This chapter presents test results for the selected materials systems. Results for earlier candidate systems are contained in Appendix A.

A. THERMAL SHOCK TESTS

The thermal shock test specimens were fabricated by bonding a 12-in. by 12-in. insulation panel to the appropriate tank metal. For the 350°F materials, the insulation was bonded to a 0.090-in.-thick plate of 2219 aluminum alloy. For the 650°F materials, the insulation was bonded either to a 0.052-in. 6Aℓ-4V ANL titanium alloy or a 0.050-in.-thick Inconel 718 plate. The edges of the metal plates were reinforced with 1-in. aluminum or stainless steel angles to prevent excessive warping.

The thermal shock test fixture is shown in Fig. IV-1. The metal side of the specimens was heated to either 350°F or 650°F with an electric radiant heater, and then the heater was deenergized and the insulation was immersed in liquid nitrogen to a depth of 1/4 to 1/2 in. The cycle was repeated after the metal plate cooled to near ambient temperature. The test fixture was designed for automatic filling and automatic cycling, and could cycle two 12-in.-square specimens simultaneously.

The cost of equipment for performing these tests with liquid hydrogen was not considered justified.

1. 350°F Insulation System

Two thermal shock specimens, shown in Fig. IV-2, were fabricated using the 350°F (350-III) insulation system. In each specimen, the facesheet was removed from three or more cells, and these cells were repaired using the method described in Section III-D.

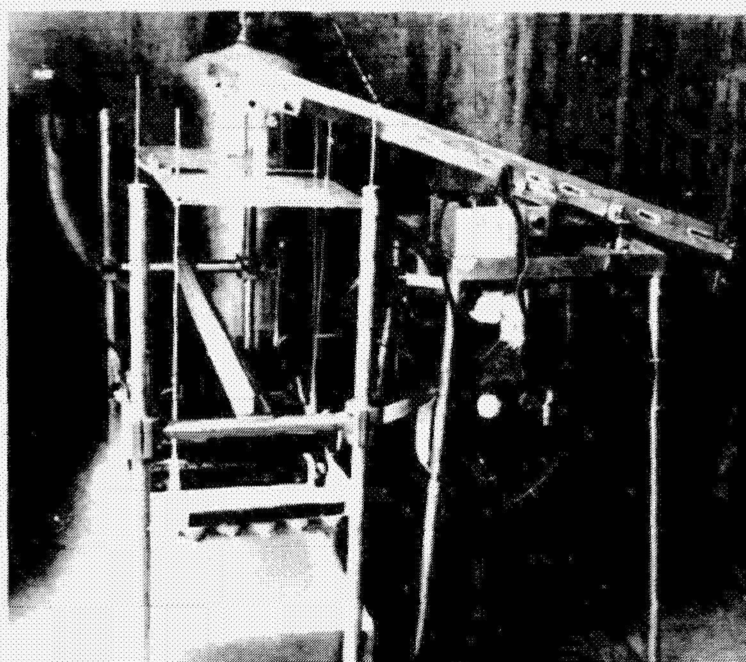


Fig. IV-1 Thermal Shock Test Fixture

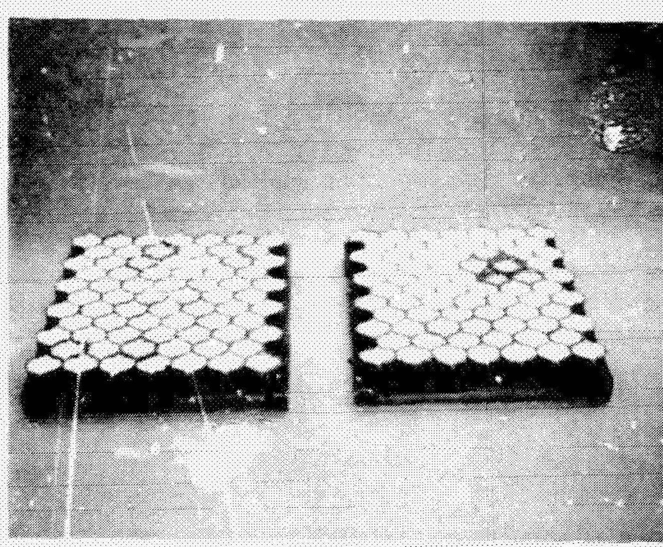


Fig. IV-2 Thermal Shock Test Specimens with Repaired Cells for 350°F Insulation System

The specimens were then installed in the thermal shock test fixture, and the aluminum plates were cycled between 70 and 350°F 308 times. No problems were apparent during the test although there was a considerable buildup of frost on the facesheets. After the thermal cycles were completed, one of the panels was found to have three tears or cuts in the facesheet. Two of the tears appeared to be due to thermal stresses, as if the Teflon had been inadequately dimpled. The dimple depth for these cells was not noticeably less than for other cells in the panel; however, the depth of all the dimples for both panels was slightly less than normal. This reduced dimple depth was due to the fact that the insulation specimens had been fabricated using tooling for panels to fit a 6-ft-diameter cylinder, and when installed on a flat panel, the facesheet was elongated. The third hole in the facesheet appeared to be a cut at the core ribbon, and could have been caused by cell pressurization due to the capillary opening being plugged with ice.

No other damage had occurred to this specimen, and none was found in the other specimen. All the facesheet patches survived the thermal cycling, and no debonds occurred in the core-to-metal adhesive.

2. 650°F System

Two 650°F insulation specimens (650-II system) were bonded to Inconel 718 plates and subjected to 196 thermal shock cycles. The Inconel sides of the specimens were cycled between 90°F and 640°F in the automatic thermal shock cycling fixture.

When the temperature of the control panel reached 640°F, the heaters were turned off and the facesheet side of the insulation was immediately immersed in liquid nitrogen to a depth of 1/4 to 1/2 in. until the plate cooled to 90°F. The total cycle time was 12 minutes (5 cycles per hour): 2 minutes were required to heat the metal from 90°F to 640°F, and it took 10 minutes to cool the plate from 640°F to 90°F.

At the peak temperature, the centers of the 12-in.-square Inconel plates were distorted in excess of 0.25 in. (in the direction of the facesheet) from their original planes. This produced much greater distortion of the insulation specimens than would be encountered in an insulated tank.

A few core-to-metal adhesive failures occurred at the points where the distortion produced high stress. Inspections revealed that these failures occurred only in areas where the core was embedded in the adhesive less than the intended 1/16-in. No damage occurred to the facesheets or to the facesheet or core node bonds.

The two thermal shock specimens are shown in Fig. IV-3. One specimen was fabricated from four separate panels to evaluate the resistance of the nested grout joint to thermal shock. There was no apparent degradation or failure of the joints, and the appearance of both panels was unchanged after testing.

B. THERMAL CONDUCTIVITY TESTS

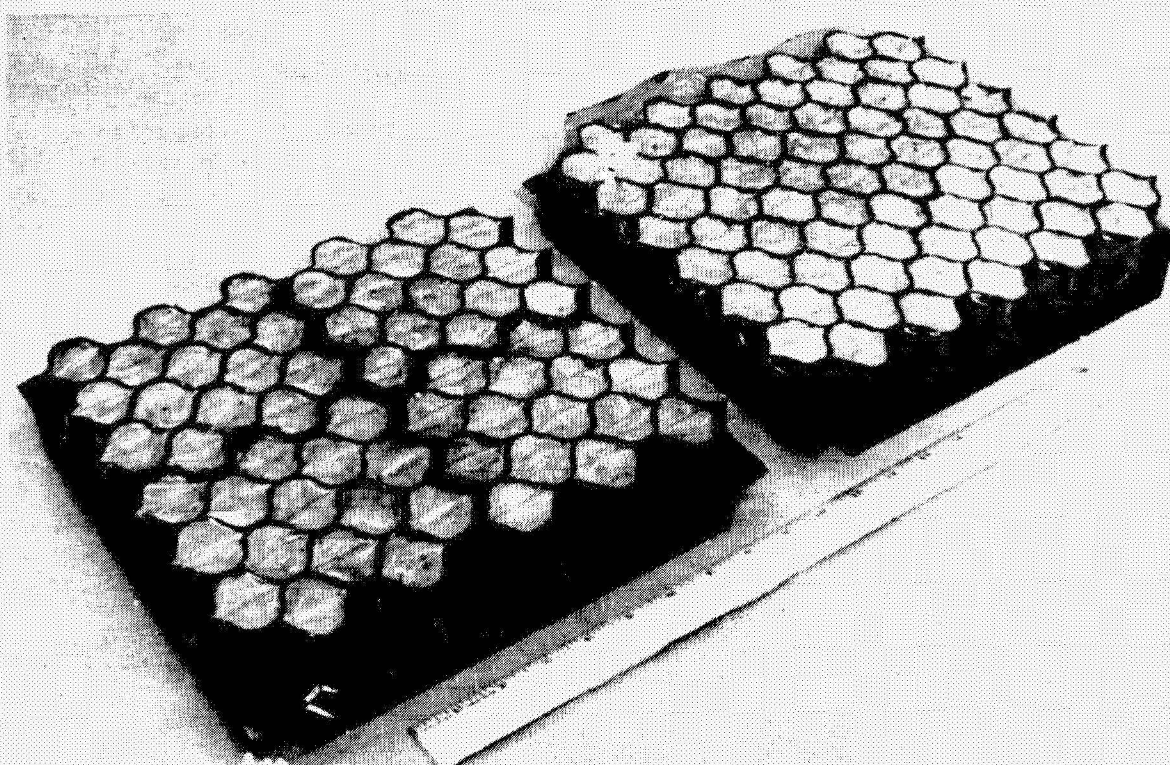
During these tests, the thermal conductivity of the insulation system was determined as a function of wall temperature. A double-sided, guarded, flat-plate calorimeter was used. Identical insulation specimens were bonded to each side of a primary and guard heater assembly, and the insulated calorimeter was submerged in liquid hydrogen.

The construction of the heater assembly is illustrated in Fig. IV-4. These units were fabricated by potting grids of resistance wire in silicone adhesive to form a thin, flat assembly, bonding plates and rings of 6061 aluminum to each side of this assembly, again using the RTV-560, and finally casting an additional ring of silicone, outside the guard rings. This outer ring isolates the guard heater from the liquid cryogen and permits the insulation panels to extend beyond the guard heater. Thermocouples were installed on the four aluminum plates and rings, and thermopiles were installed between the primary plates and guard rings on each side.

The primary heater was 8 in. in diameter, the guard heater had an OD of 14 in., and the complete assembly was 17 in. in diameter. Figure IV-5 shows the calorimeter assembly prior to installing the insulation panels.

The double-sided, flat-plate calorimeter described above replaced a square calorimeter assembly, which is described in Appendix A. Modifications that were made to this earlier device to prevent excess heat transfer from the guard heater to the cryogen increased its overall dimensions to the point where it could not be installed horizontally in the test dewar.

The thermal conductivity tests were conducted using a method similar to that described in ASTM Standard Method C-177. The insulated calorimeter assembly was installed in the dewar by suspending it from the cover assembly. The calorimeter was installed both horizontally and vertically to evaluate the effect of the orientation with respect to gravity on the performance of the insulation.



Fi. 'nconel Thermal Shock Test Specimens

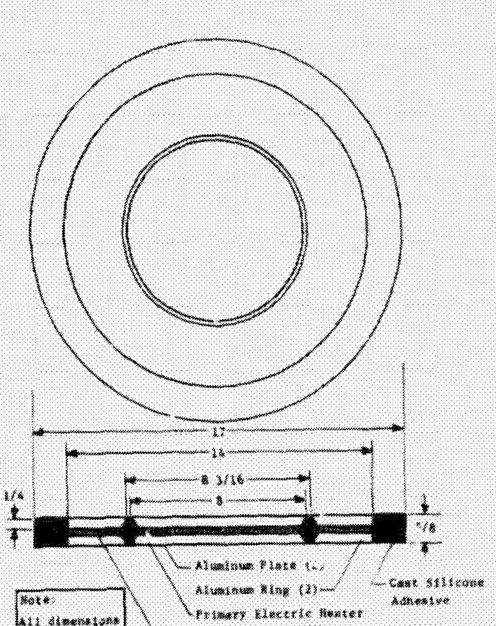


Fig. IV-4 Calorimeter Heater Assembly

Fig. I'-5 Circular :lorimeter before Installing Insulation

The dewar and the insulation were purged by alternately pressurizing the container with helium and then venting. After purging, the vessel was filled with liquid hydrogen and held to a level above the test specimen. Power was applied to raise and/or hold the temperature of the primary heater to the desired test value. An automatic control system provided power to the guard heater so as to control the temperature difference between the primary and guard heaters, as sensed by the thermopile, to within less than 1/2°F.

The combined heat flux through the primary areas of the insulation on both sides of the calorimeter was determined from the power input to the primary heater. The thermal conductivity was then determined from this heat flux and from the effective area of the primary heater, the thickness of the insulation, and the temperature difference across the insulation by using the relationship

$$k = \frac{QX}{2A\Delta T}$$

where

k = measured thermal conductivity, Btu/hr-ft-°R

Q = heat input rate, Btu/hr

X = insulation thickness, ft

A = effective area of one side of primary heater plate, ft^2

ΔT = difference between measured temperature of primary plate and temperature of saturated cryogen at test pressure, °R.

The circular calorimeter was insulated with the 350-III materials system (described in Chapter I), which included Kapton core, Teflon facesheet, and nonopacified fiberglass filler (see Fig. IV-6).

Nonopacified filler was chosen because it was considered adequate for the reusable boost tank application, where the insulation effectiveness at high wall temperatures is of secondary importance. For walls at or below ambient temperatures, radiation heat transfer was not expected to be significant in comparison with gas conductivity. No special effort was made to dry the filler material before it was installed.

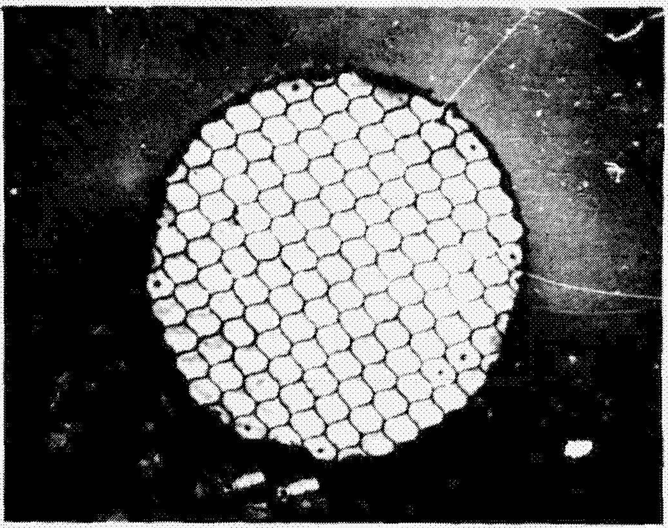


Fig. IV-6 Circular Calorimeter with 350-III Materials System

The results of thermal conductivity tests using this specimen are shown in Fig. IV-7. Test data for horizontal and vertical orientations are given. The figure also shows the results from a prior test using the square calorimeter in the vertical orientation, and the integrated or effective thermal conductivity of gaseous para hydrogen. The effective thermal conductivity of hydrogen gas between the temperature of the liquid and any other temperature, T, is defined by

$$k_{(T,\ 36)} = \frac{\int_{36}^{T} k(T)\ dT}{T - 36},$$

where

$k_{(T,\ 36)}$ = effective thermal conductivity of a layer of hydrogen gas with boundary temperatures of 36°R and T (°R)

k(T) = the thermal conductivity of gaseous para hydrogen at any temperature, T, as given by WADD Technical Report 60-56.*

**A Compendium of the Properties of Materials at Low Temperature (Phase I). Part I - Properties of Fluids.* WADD Technical Report 60-56 (Contract AF33(616)-58-4). Wright Air Development Division, Air Research and Development Command, Wright-Patterson AFB, Ohio, October 1960.

The dashed portion of the curve has been extrapolated beyond the data given in the reference. The curve for the thermal conductivity of hydrogen represents the theoretical lower limit, or best possible performance, for the capillary internal insulation system.

The curve for the previous test with the 350-II system follows the slope of the hydrogen conductivity curve more closely than that for the 350-III system. This is due to the difference in filler material. At the higher temperatures, heat transfer by radiation becomes significant for the 350-III system with the nonopacified filler material. However, for temperatures between 350°R and 400°R, which might be expected during a ground hold, the two systems are approximately equivalent and have thermal conductivities from 27 to 38% greater than that for gaseous hydrogen.

Using a wall temperature of 530°R (70°F), and assuming an average density for 1-in.-thick installed insulation the product of density and thermal conductivity ranges from 0.305 to 0.357 Btu-lb/hr-ft^4-°R.

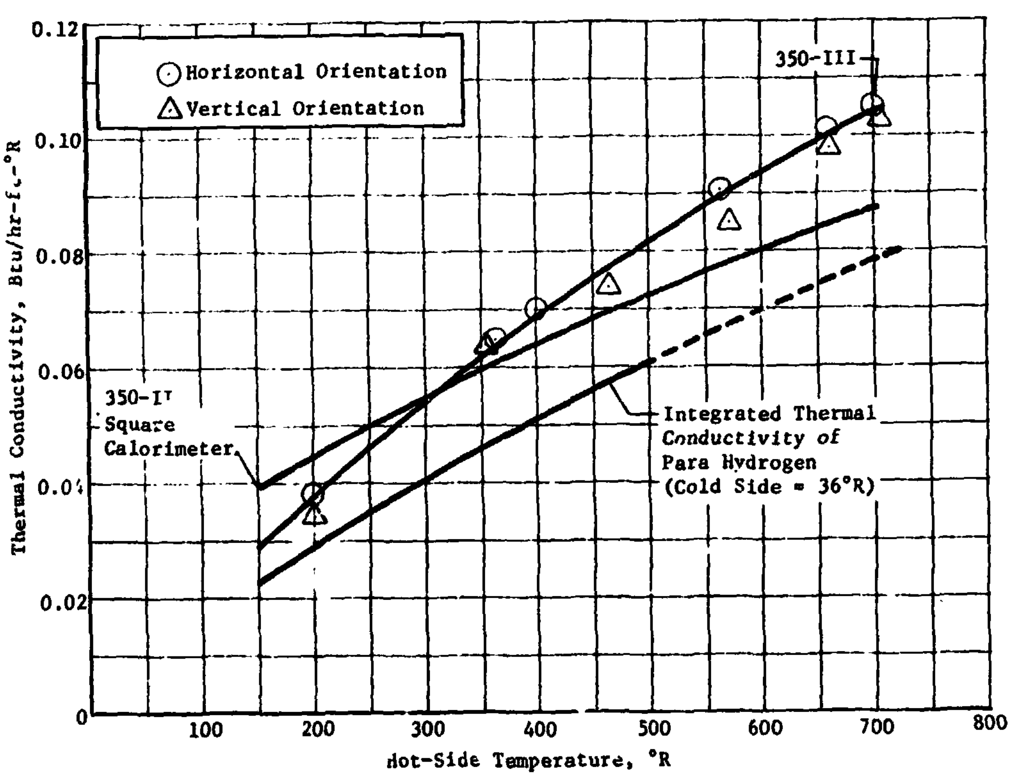


Fig. IV-7 Thermal Conductivity Test Results

C. DOME CYCLING TESTS

Dome cycling tests were performed to evaluate the ability of insulation fabricated from various materials systems to withstand temperature and pressure cycles when installed as part of a hydrogen tank. The setup for these tests is shown in Fig. IV-8.

The insulation was bonded to the inside of a 19-in.-diameter metal dome and installed at the bottom of the dome cycling test fixture (see Fig. IV-9). After being purged with helium, the chamber was filled with liquid hydrogen and pressurized (to either 35 or 45 psig). The dome was then heated using external radiant heaters to either 350°F or 650°F. After the dome reached the desired temperature, the heaters were turned off and the pressure was released to ambient. The dome was cycled repeatedly from ambient pressure and temperature to the operating pressure and temperature (either 350°F or 650°F).

The barrel of the dome cycle test fixture was insulated with a low-temperature materials system. This capillary internal insulation was fabricated from Nomex core, Teflon facesheet, polyurethane adhesives, and polystyrene filler. It survived quite well and continued to serve the purpose of reducing hydrogen usage throughout the test program.

For the 350°F insulation system, we used a 2219 alimunum alloy dome. A 0.052-in. titanium (6Al-4V ANL) dome was used for the initial 650°F insulation system; results of test with this dome are presented in Appendix A (650-I system), along with results of dome cycle tests for the 350-I and 350-II systems. An 0.050-in. Inconel 718 dome was used to evaluate the 650-II silicone adhesive system.

The domes used in these tests were explosively formed at our High-Energy Forming Facility. The aluminum dome was chemically milled to a thickness of 0.032 in. This resulted in stressing the dome to a safety factor of only 1.8, based on its yield strength at 350°F, when the pressure was 45 psig.

Because of the inadequate pressure capability of the test fixture and the higher yield strengths of titanium and Inconel 718, we were unable to achieve this 1.8 safety factor when pressurizing the other domes.

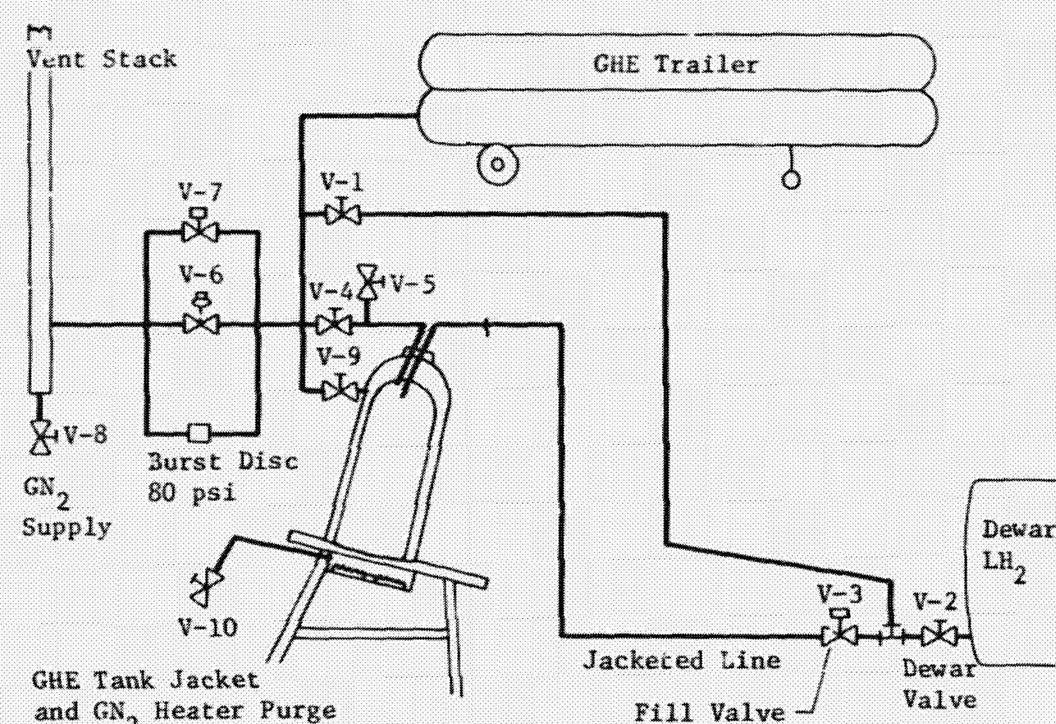


Fig. IV-8 Setup for LH_2 Dome Cycling Test

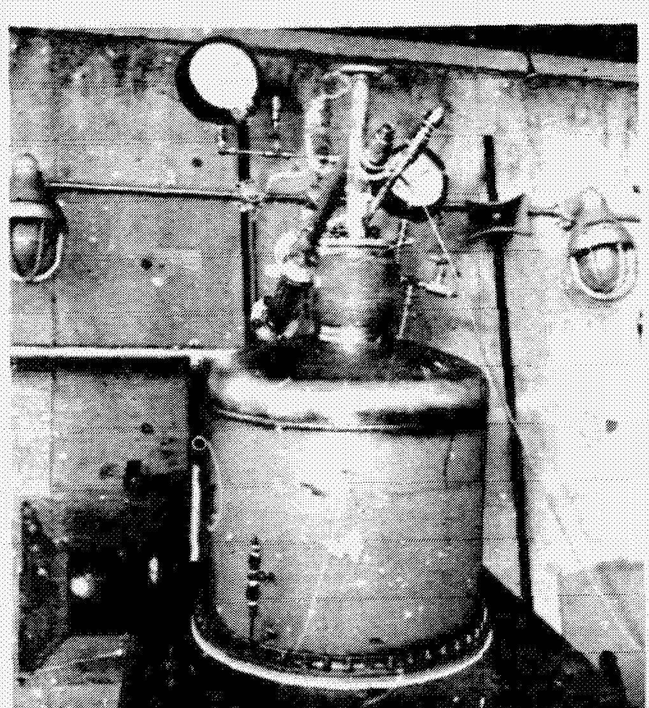


Fig. IV-9 Test Fixture for Dome Temperature and Pressure Cycle Tests

1. 350°F Aluminum Dome

The aluminum dome was insulated with the 350-III system using the fabrication method described in Chapter III. The design of this specimen was based on the radiating ribbon concept, and two semicircular panels were fabricated for it.

To evaluate the stencil adhesive application technique, we installed one of the panels by applying adhesive continuously to the dome section, and installed the other panel by using the stencil to apply the adhesive. After fabrication, we found that several pairs of cells were interconnected. In such cases, we sealed the opening in the facesheet for one of the cells.

This dome specimen was then installed at the bottom of the test vessel. After pressure purging the vessel with helium, it was filled with liquid hydrogen at a maximum rate with no delay for cooldown. The pressure was then increased to 35 psig* at a rate of approximately 20 psi per minute. Power was then applied to the external radiant heater, and maintained until the thermocouple near the center of the dome reached 350°F. Heater power was then turned off, and the pressure was released at approximately 20 psi per minute. The next cycle was initiated by refilling the vessel with liquid hydrogen.

The dome specimen was subjected to 23 cycles before it was removed, inspected, and found to have failed. Failure occurred when the insulation debonded from the dome over approximately 75% of the total area. No damage had occurred to the Teflon facesheet or to the silicone adhesive bond between the facesheet and the Kapton core. However, some damage had occurred to the core at the outer perimeter. This damage was adjacent to several areas where an excess of adhesive had accumulated, but may have occurred while installing the dome in the test fixture. The core node bonds (made with RTV-156 adhesive) had remained intact except for small areas of peel failure that had occurred in conjunction with the debonding at the metal interface. The RTV-560/Cab-O-Sil grout joint had remained intact.

---

*This test was conducted at 35 psig, instead of the 45 psig that had been used in previous tests because we felt the aluminum dome might have been weakened by the repeated cleaning of previous candidate adhesives from the dome.

This specimen was assembled shortly before the silicone bonding and primer problems (described in Section II-B) were encountered. Consequently, its debonding was probably related to the primer curing problem. Since we had previously obtained excellent core-to-aluminum bonds using RTV-560, this specimen was rebuilt. The reinsulated test article is shown in Fig. IV-10.

The same materials were used, but the improved primer and adhesive curing procedure described in Chapter III was incorporated in the fabrication process. Sample tests of the silicone-to-metal bond were evaluated and found to be satisfactory before the insulation panels were installed in the dome. As before, adhesive was stenciled on one-half of the aluminum dome and spread continuously on the other half. The dome adhesive was allowed to cure for 10 days.

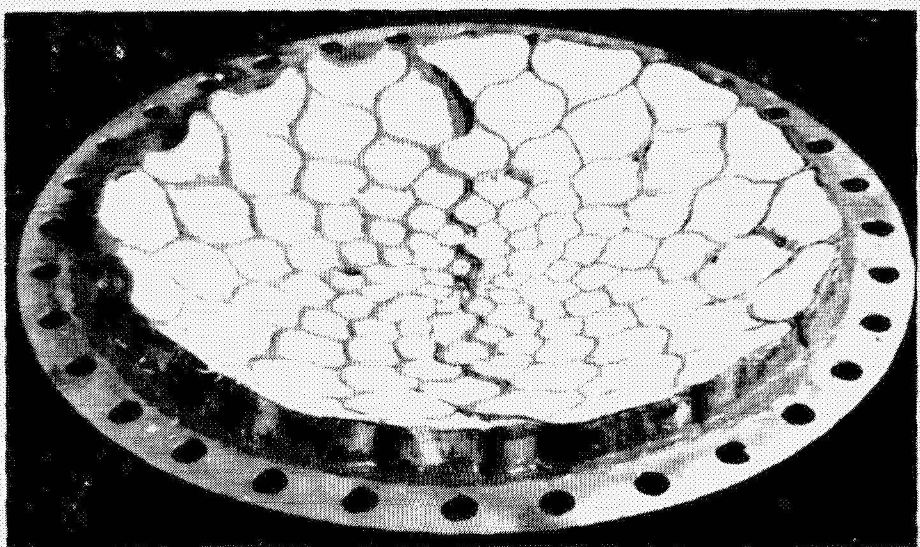


Fig. IV-10 Aluminum Dome Insulated with 350-III Materials System

The rebuilt dome specimen was cycle-tested using the same procedure described above for the previous dome. A total of 24 cycles was achieved, using one 1500-gallon supply of liquid hydrogen. No failure occurred in the core-to-dome bond, indicating that the previous debonding was probably related to the primer curing problem. The grouted joints also remained intact. However, several tears occurred in the Teflon facesheet, and one outer cell experienced damage that was known to have occurred during installation in the test fixture.

A detailed posttest examination of the specimen indicated that the facesheet tears were related to cells that were somewhat distorted in shape. We also noted that a tear in the Kapton core ribbon was associated with each facesheet tear. Attempts to create similar failures in other cells at room temperature and at -320°F were unsuccessful.

We concluded that the most likely cause of the failures was small unnoticed notches that had been created in the core ribbons when they were originally being trimmed. The combination of these notches and the distorted cell shape probably was required to cause the failure. Both represent defects that would not occur if production tooling were used.

2. 650°F Inconel Dome

The Inconel dome was insulated using four pie-shaped panels, plus a small, star-shaped, single cell in the center. All panels were fabricated from the 650-II materials. The radiating ribbon design approach, with nested grout joints between the panels, was again used. Figure IV-11 shows the four panels before installation, and Fig. IV-12 shows the small star-shaped cell bonded in place. The completed dome is shown in Fig. IV-13.

Smaller panels were used for this specimen to minimize the degree of distortion required to comform the facesheet to the required compound curvature. To further minimize this problem, the core was expanded onto a flat rake that was designed to achieve the required panel size and shape after it was formed to the curvature of the dome. The facesheet was then bonded in place in a flat plane, and no preforming was required.

The core was made from curved ribbons that were designed to expand to the proper curvature. Consequently, when the bonded assembly was removed from the rake, it assumed a curved shape very near the required final shape. This technique was only used in this one application, yet it appears to be an acceptable simplification of the bonding process for panels with a high degree of compound curvature.

The panels were installed onto the dome without using tooling to control their position, and the adhesive was applied over the entire surface. Because of difficulty in controlling the position of the panels, and because of inaccuracies in the design, the grout joint exceeded 1/2 in. in some places.

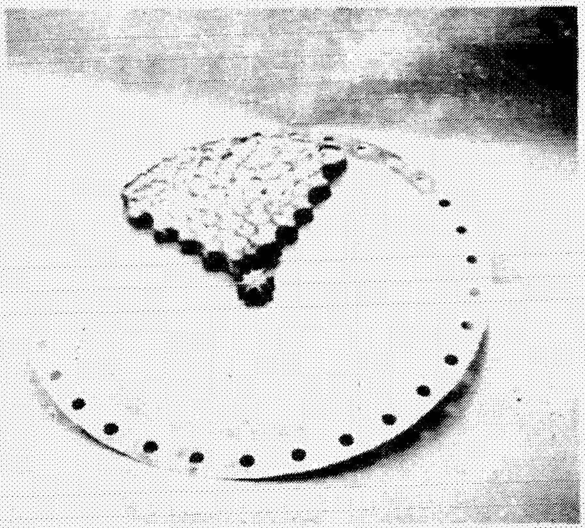


Fig. IV-11 Insulation Panels for Inconel Dome

Fig. VI-12 Inconel Dome after Installation of Center, Star-Shaped Cell

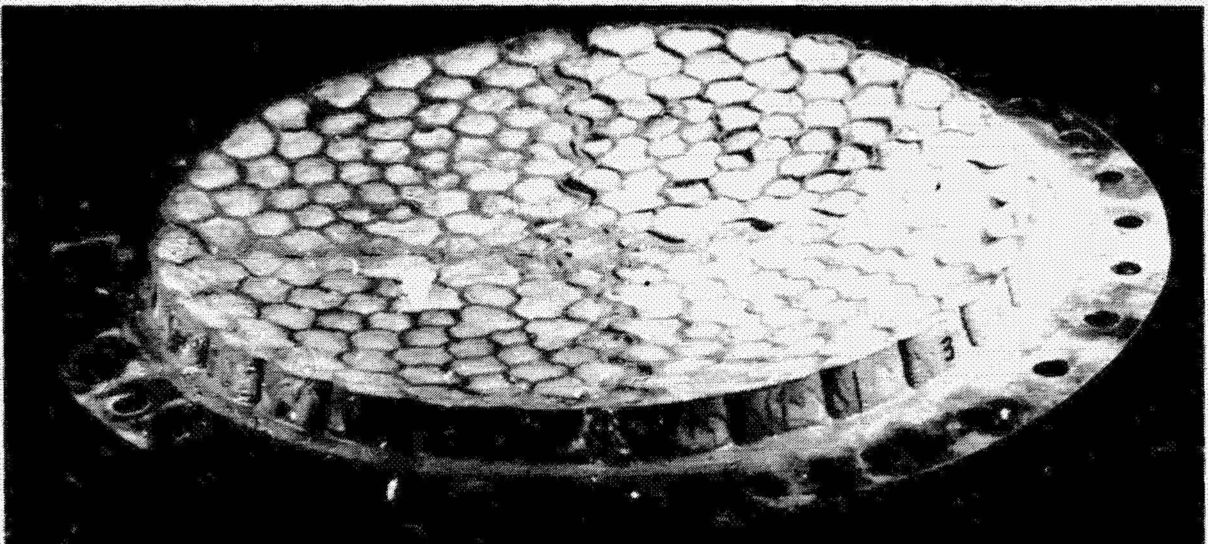


Fig. IV-13 Completed Inconel Dome

The dome was installed in the dome cycle fixture and the test vessel was purged with helium. Temperature and pressure cycles were accomplished by (1) filling the vessel with liquid hydrogen, (2) applying power to the external heater to raise the dome temperature to 650°F, (3) holding the dome temperature at 650°F while increasing the internal pressure to 45 psig, and (4) deenergizing the heater and venting the pressure. The cycle was repeated after the dome had reached ambient temperature or below.

During the eighteenth cycle, the temperature measurements indicated an insulation failure at the center of the dome. The tests were stopped after the nineteenth cycle was completed. A posttest inspection of the specimen revealed that the facesheet had torn in and near the center star cell. It was readily apparent that these tears had propagated from breaks in the grouting between the panel. These breaks had occurred at three places where the joint was still very narrow, but near extremely wide sections of grout. (This condition would not occur in an actual tank installation, where more adequate design and tooling are provided.) No other damage to the insulation was found.

V. CONCLUSIONS

The capillary gas-layer internal insulation concept developed on this program can be applied to reusable-booster liquid hydrogen tanks that will repeatedly experience high temperatures due to reentry heating. Materials systems have been selected and demonstrated to be suitable for repeated exposure to tank wall temperatures of 350°F and 650°F in a hydrogen environment. Insulation design concepts were selected, developed, and demonstrated in tests on small specimens.

The thermal conductivity of the insulation system was measured for various wall temperatures using liquid hydrogen as the operating fluid. The system's performance as thermal insulation was found to be near the expected range, and totally adequate for the liquid hydrogen boost tank application. System tests using small specimens demonstrated the capability of the insulation to withstand repeated thermal shock and combined temperature and pressure cycling.

System design and fabrication processes were evolved to a considerable extent on the basis of failures experienced in system tests. This development cycle demonstrated that the insulation could survive the extreme operating environments and maintain satisfactory performance, even when made of relatively weak materials, so long as the necessary design and fabrication requirements were met. However, certain design and fabrication deficiencies were identified that could cause failure in the insulation system. The design criteria, tooling concepts, fabrication methods, and quality requirements that were developed have eliminated these failures, and when applied to the insulation of large tanks should result in insulation systems with consistently predictable performance, long life, and high reliability.

Although a wide variety of materials was initially investigated, the materials finally selected for the 350°F and the 650°F insulation systems differed only in the film used for the facesheet. Initially, difficulty was experienced in satisfactorily dimpling the Kapton facesheet used in the high-temperature systems and Teflon was selected for the facesheet for the lower-temperature insulation. Only near the end of the program did we develop a completely acceptable method for dimpling the Kapton facesheet. Since the 1-mil Kapton facesheet is significantly lighter than the 2-mil Teflon facesheet, it might prove to be a better choice for 350°F applications.

It is difficult to accurately project the cost of fabricating and installing the capillary internal insulation system on the basis of the small-scale specimens that were fabricated during this program. Our experience indicates that the cost of materials for the selected systems, including installation materials, will be in the range of \$3.50 to \$4.00 per square foot for a thickness of 1 in. The direct labor required for panel prefabrication should be 1.25 hr or less, and panel installation should require no more than 0.5 hr per square foot.

The weight of the system is approximately 1/3 lb per square foot of installed insulation for a 1-in. thickness, or 4 lb/ft^3. Since about 2/3 of the weight is in the facesheet assembly and the installation adhesive, the insulation density would decrease for increased thickness, and would increase if less than 1 in. were used.

The 350°F insulation system, along with design and fabrication data that were developed on this program are being used to insulate a 6-ft-diameter by approximately 7-ft-high aluminum tank under Contract NAS3-14384, *Internal Insulation System Development*, for NASA's Lewis Research Center. This tank will be tested with liquid hydrogen and will be subjected to thermal cycling to a temperature of 350°F. These tests will demonstrate the performance of the insulation and its ability to withstand the high-temperature environment when applied as a complete system. Additional effort that will be required to apply this insulation system to space vehicles will include developing high-quantity production tooling and procedure, and testing insulation produced in quantity when installed in larger test tanks.

APPENDIX A

ADDITIONAL TEST DATA

In addition to the adhesive systems discussed in Chapter IV, also evaluated several other systems on the basis of materials testing results obtained early in the program. These materials systems are designated 350-I, 650-I, and 350-II. The evolution of our adhesives selection criteria is discussed in Chapter II.

The following sections describe the results of thermal shock, thermal conductivity, and dome cycling tests using the various adhesives combinations.

A. 350-1 INSULATION SYSTEM

Based on the results of our material evaluation in Task I, we initially selected the following materials for the 350°F insulation system.

Core	5-mil Kapton Film
Facesheet	2-mil Teflon Film
Core Node Adhesive	Thermadite 17
Facesheet/Core Adhesive	Lefkoweld 109/LM-52
Core/Aluminum Adhesive	Lefkoweld 109/LM-52
Filler Material	Owens-Corning PF-105-700 T (opacified fiberglass)

1. Thermal Shock Tests

Two sets of two each 350-1 thermal shock test specimens were used. The first set debonded on approximately the third cycle. After the second set underwent four cycles, a visual inspection indicated that the facesheet on one cell had broken. During the 46th cycle, the temperature control mechanism failed and the panels were subjected to approximately 900°F. Only one panel severely debonded. The other panel was cycled 17 additional cycles without failure before the test was stopped.

An additional small test panel was bonded to an aluminum panel with Lefkoweld 109/LM-52 and placed in the thermal shock device. At 350°F the Kapton core was pulled away from the aluminum plate with only a small amount of force. Due to the reduced strength and softness of the adhesive at 350°F, we decided not to proceed with any further thermal shock tests with this materials systems.

2. Thermal Conductivity Tests

A calorimeter assembly, shown in Fig. A-1, was fabricated using the 350-I materials system. After several hours of testing with liquid nitrogen, failure points were evident around the edge of the guard heater and around power leads, thermopile leads, thermocouple leads, and support points. Although repairs were made and testing was again attempted, satisfactory results were not achieved with this device.

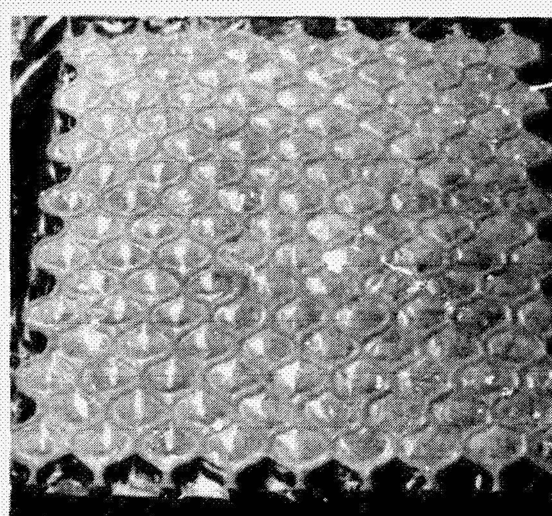


Fig. A-1 350-I Insulation Installed on Square Calorimeter

B. 650-I INSULATION SYSTEM

The materials we initially selected for the 650°F insulation system were as follows:

Core	5-mil Kapton Film
Facesheet	5-mil Kapton Film
Core Node Adhesive	Thermadite 17
Facesheet/Core Adhesive	BR-34
Core/Tank Wall Adhesive	BR-34
Filler Material	Owens Corning PF-105-700 (Opacified fiberglass)

This system was tested with titanium as the candidate tank material.

1. Thermal Shock Tests

The 650-I system was subjected to 300 thermal shock cycles from 650°F to -320°F. Figures A-2 and A-3 show the two 650-I thermal shock test specimens. Each panel had 50 complete cells. The facesheet of one cell failed on the first cycle. No other failures in the facesheet or core bond were detected after 300 cycles.

2. Thermal Conductivity Tests

Figure A-4 shows the 650-I insulation installed on the calorimeter. Thermal conductivity measurements with this device greatly exceed the predicted value. An inspection showed that there were no major failure points; however, leakage between the cells appeared to have caused the high values. Leakage through voids created by volatiles in the polyimide adhesives was a major problem with this system.

Because of the unsatisfactory thermal conductivity tests on the first 650-I calorimeter, several test coupons were fabricated with the polymide adhesives FM-34, BR-34 and T-17 in an attempt to eliminate the voids between cells. These coupons were leak-checked under water to determine which techniques gave the most satisfactory results. As a result of this investigation, the build procedure was reversed: a vacuum bag was used to load the core while bonding it to the metal, and the Thermadite-17 adhesive, rather than the BR-34 adhesive, was used. In the reversed build procedure, the core was bonded to the metal plate; the bonds were leak-checked; leaks were repaired; fiberglass was placed in the cell; and the facesheet was bonded to the honeycomb. Although this method produced some improvement, there were still an unsatisfactory number of leaking cells and no further calorimeter tests were conducted. Subsequent development of bonding techniques with polyimide adhesives is described in Chapter III.

3. Dome Cycling Tests

The 650-I dome specimen failed on the initial cycle with liquid hydrogen. A posttest inspection showed that there was a complete loss of dimpling in the facesheet, and facesheet failure in several places. The dimple loss was initially attributed to a lack of Kapton preshrinking; however, we later discovered that the dimples had not been heat-set at a sufficiently high temperature.

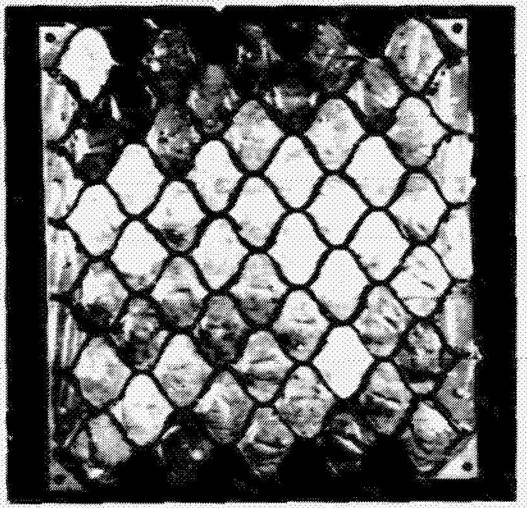


Fig. A-2 650-I Thermal Shock Test Specimen #1

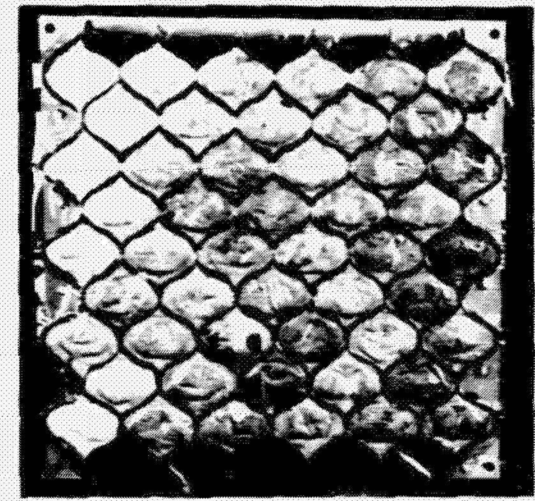


Fig. A-3 650-I Thermal Shock Test Specimen #2

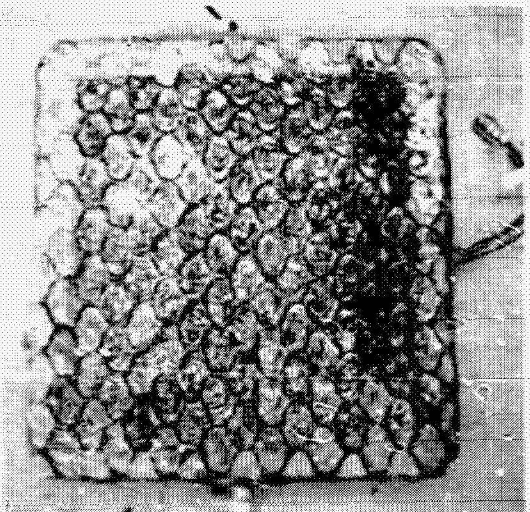


Fig. A-4 Calorimeter with 650-I Insulation Installed for Thermal Conductivity Tests

C. 350-II INSULATION SYSTEM

After experiencing bonding failures with the 350-I and 650-I insulation systems, we reviewed vendor literature on silicone adhesives to determine whether this family of adhesives was satisfactory for use in the insulation system. Some preliminary tests were conducted with silicones and primers on Teflon and Kapton in liquid nitrogen for indications of debonding. Since the results of these tests were encouraging, we selected the following materials for the 350°F insulation system.

Core	5-mil Kapton Film
Facesheet	2-mil Teflon Film
Core Node Adhesive	Thermadite 17
Facesheet/Core Adhesive	RTV-560
Core/Aluminum Adhesive	RTV-156
Filler Material	Opacified Fiberglass
Primer on Core and Facesheet	SS-4155
Primer on Aluminum	None

1. Thermal Shock Tests

The 350-II thermal shock specimens successfully completed 300 thermal shock cycles. The panels were leak-checked before and after the test series by using the nitrogen flowrate indicator and regulator. No cell failures could be detected. Figure A-5 shows one of the 350°F thermal shock specimens after the test.

2. Thermal Conductivity Tests

The 350-II insulation system was bonded to the calorimeter as shown in Fig. A-6. The edge around the calorimeter was filled with RTV-560, which made a seal around the electrical leads and structure support points. The half cells around the edge of the calorimeter insulation were closed out with a straight piece of 5-mil Kapton film. The calorimeter specimen was tested using liquid hydrogen as the test cryogen. Tests were conducted with the insulation in the vertical plane only.

The calorimeter test was accomplished without any mechanical problems. Test data were obtained for hot-side temperature from 170°R to approximately 670°R. These results are shown in Fig. A-7.

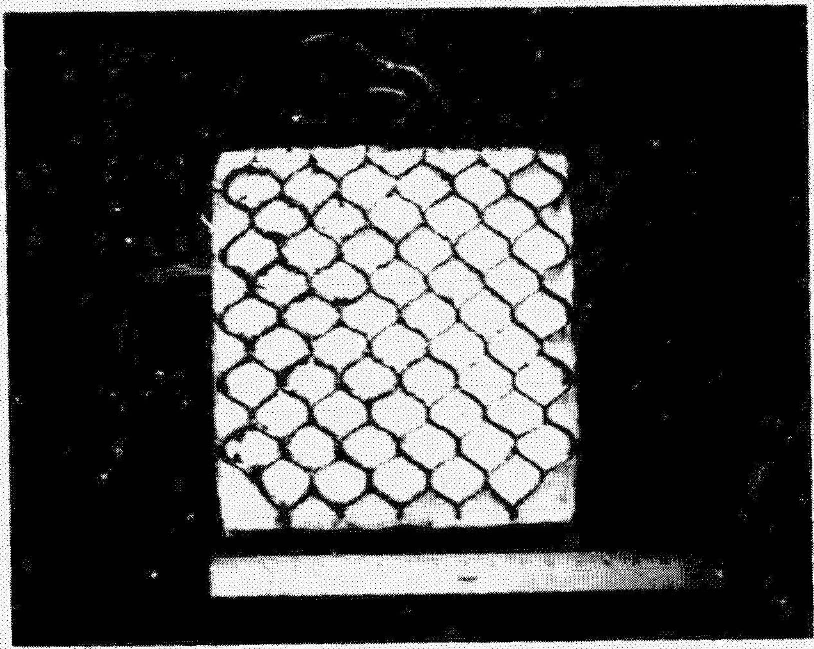


Fig. A-5 350-II Insulation Thermal Shock Panel

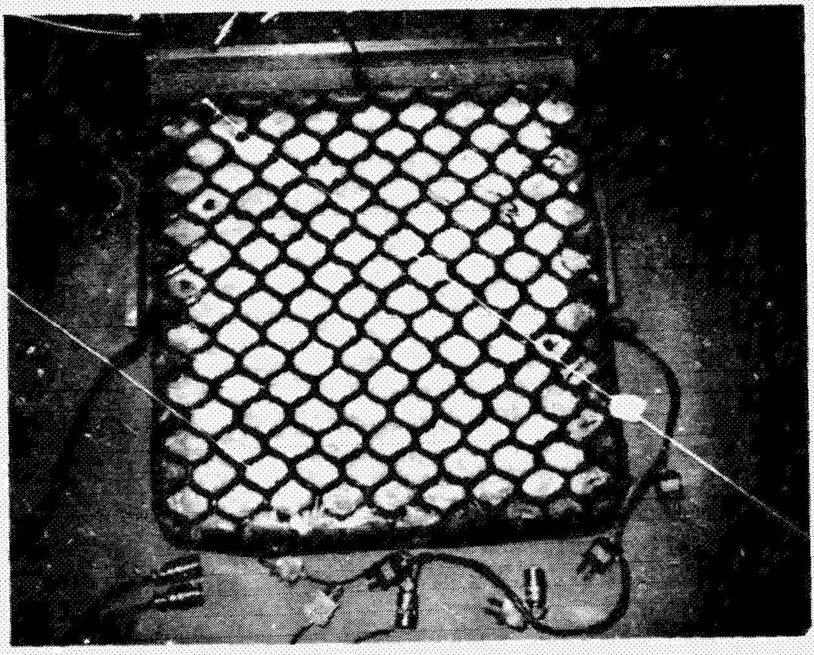


Fig. A-6 350-II Thermal Conductivity Test Specimen Mounted on Calorimeter

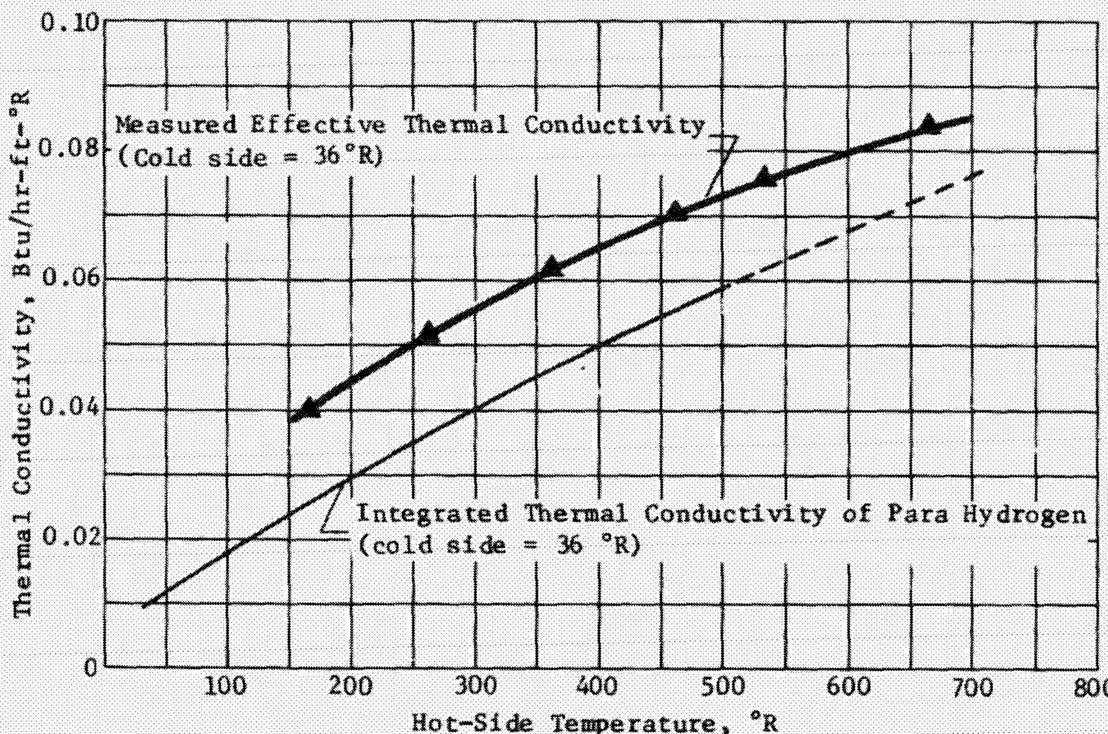


Fig. A-7 Calorimeter Thermal Conductivity Test Data for 350-II Materials System

Figure A-7 also shows the effective or integrated thermal conductivity of gaseous para hydrogen. At higher temperatures, the thermal conductivity was approximately 17% greater than that for gaseous hydrogen. At lower temperatures, the deviation was approximately 60%. No failures were apparent in the insulation, and based on other tests, we expect the heat transfer by radiation through the opacified fiberglass to be negligible in this temperature range (when compared with conduction through hydrogen gas).

The only known anomaly in the insulation system was the excessive compression of the fiberglass filler material. Because of this compression, approximately 10% of the cell on the facesheet side was not filled.

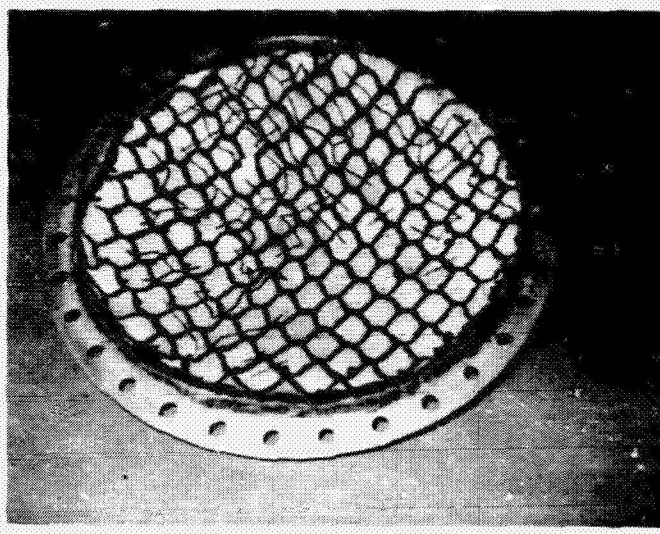


Fig. A-8 350-II Dome Cycling Test Specimen

3. Dome Cycling Test

The 350-II insulation system is shown installed in the aluminum dome in Fig. A-8. The dome was tested with liquid hydrogen and was pressurized to 45 psig five times without the heater installed. Each time the thermocouples on the exterior side of the dome indicated a colder temperature. It is normal for the temperature to drop when pressure increases, since liquid is forced through the capillary opening and has a cooling effect until it is vaporized; the temperature will then recover until a steady-state condition is again achieved. However, in this test, the temperature did not recover significantly. The test was terminated and the fixture was allowed to come to ambient temperature.

After the test, we found that the dome specimen had partially debonded on the outer edges between the core and dome. Since the lower dome is removable, there is a gap where liquid hydrogen can contact the bond between the dome and the core. Debonding apparently began around the edges and propagated toward the apex of the dome.

Later test results indicated that the dome failure may have been due to incomplete curing of the RTV-156 adhesive. A relatively thick layer of adhesive was used to bond the core to the aluminum. The thickness of the adhesive and the enclosed location of the bond area may have prevented the exposure to air required for curing. Since curing RTV-156 for such interior bonds presents a problem, this adhesive was eliminated for the core/aluminum bond.

APPENDIX B

FABRICATION PROCESS SPECIFICATION

The following process plan describes the steps required to fabricate insulation panels for tanks with simple and compound curvature. Type A panels are rectilinear-core panels for insulating sections with simple curvature (cylindrical or barrel sections of a tank). Type B panels are ring-segment panels with a radiating-core-ribbon pattern for insulating sections with compound curvature (domes or a transition between a dome and cylinder).

The special tools required for fabrication are listed in Table B-1. The "X" in the tool number refers to the designation of the specific panel for which the tool is designed. Panel and tool dimensions would be specified on the detailed panel drawing.

Table B-1 Fabrication Tooling

Tool No.	Description
TX01	Drill Template
TX02*	Short Strip Trim Template
TX03	Print Stencil
TX04	Stacking Press
TX05	Outline Trim Template
TX06	Assembly Tool (Assembly Rake/Vacuum Box)
TX10	Dimpling Vacuum Box
TX12	Contour Form
TX14	Vacuum Box Lid (Carrier Frame)
TX15	Transfer Sheet (Rubber or Polyethylene)
TX16	Expansion Tool
TY17†	Filler Plug Die Sets
TY18	Filler Plug Insertion Tool
T001	Dimple Depth Tool

*This tool is used only for panels that have both short and long core ribbons.

†The "Y" corresponds to the filler plug designation, which may not correspond to the designation of the panel(s) in which it is used.

DATE (REV.) 5-9-72

S/A IDENT. ______ STEP 5 SHEET 1 of 2

SUBSTEP	OPERATION
	This step is for assembling the core for Type A or Type B panels. Tools needed for this step are a paper cutter, water basin, drill and No. 30 drill bit, bandsaw and special no-set fine-toothed blade, print frame, scissors, scraper blade, marker pen, drill template (TX01), short strip trim template (TX02) for B panels, print stencil (TX03), stacking press (TX04), trim template (TX05), expansion tool (TX16), oven, and humidity room. Materials needed for this step are rolls of 5-mil-thick Kapton film in the required widths, cheesecloth, gaseous nitrogen, RTV-156 adhesive, and core assembly storage boxes.
a.	Using the paper cutter, cut the required number of Kapton strips in accordance with the panel drawing.
b.	For B panels, align the Kapton strips between the halves of the drill template (TX01). Clamp the halves together. Drill the positioning holes as indicated by the template. Remove the strips from the template. For A panels, proceed to Substep d.
c.	For B panels,* position the short strips between the halves of the short strip trim template (TX02) and clamp the halves together. Using the band-saw and special blade, trim the end of the strips marked "TRIM." Remove the strips from the template halves.
d.	Clean the Kapton strips with trichlorethylene.
e.	Place the strips in the water basin and soak them for a minimum of 12 hr.
f.	Insert the print stencil (TX03) in the printing frame.
g.	Remove the Kapton strips from the water trough and dry them with cheese-cloth. Place the dry strips side by side on cheesecloth spread on top of the workbench.
h.	Start the nitrogen purge in the print frame and add enough RTV-156 to one end of the stencil to finish stenciling the required number of strips for one panel.
i.	Place a strip in position on the alignment pins in the print frame. For A panels, align the strip end with the "START" line and perforate the strip with the punch. For B panels, refer to the appropriate panel drawing to find the sequence for perforating the short and long strips.
j.	Apply the RTV-156 to the strip through the specified openings of the stencil (in accordance with the applicable panel drawing), using the scraper blade to apply and remove excess adhesive.
	*This step trims the ends of short ribbons, which are inaccessible after assembly.

DATE (REV.) 5-9-72

S/A TDS. ?. STEP 5 SHEET 2 of 2

SUBSTEP	OPERATION
k.	Remove the printed strip from the print frame and place it, adhesive-side up, on the alignment pins of the stacking press (TX04). For A panels, the first strip is indexed in the "A" position (see appropriate drawing), the second in the "B" position, and each subsequent strip is alternated. For B panels, refer to the panel drawing.
ℓ.	Repeat Substeps j thru ℓ until all but the last strip has been printed.
m.	Place the unprinted strip in the stacking press on the alignment pins. For A panels, punch the last strip (Substep j) and place it in the stacking press in the "A" position.
n.	Place the top half (pressure bar) of the stacking press on the alignment pins and clamp the halves together at the preset stop. (Note that more than one core assembly can be pressed at one time if the correct preset stop is used to account for the thickness of the additional assemblies.)
o.	Place the stacking press in the humidity room and leave the assembly in position for a minimum of 12 hr cure the adhesive.
p.	Remove the core assembly from the stacking press and leave it in the humidity room for a minimum of 72 hr.
q.	Expand the core on the expansion tool (TX16) and visually inspect the assembly for bondline quality. If any bondline is defective or questionable, the discrepancy shall be noted on a tag and attached to the core. The assembly will be reviewed by the program manager for final disposition. Remove the core from the expansion tool.
r.	Place the core assembly in the outline trim template (TX05) and clamp the template halves together. For A panels, place the strip assembly between the guide pins of the template, with the "TRIM" end of the template at the center of one of the strip-end bondlines and with the template windows centered over the adjacent bondline. Trim the outline of the core using the bandsaw equipped with the special blade. For A panels, only the ends of the core are trimmed. Inspect the trimmed edges.
s.	Heat the oven to 450°F, place the unexpanded core assembly in the oven, and postcure the adhesive at 450°F for a minimum of 4 hr.
t.	Turn off the oven, remove the core, and allow the core to cool to room temperature.
u.	Apply the ID number to the core with the marker pen.
v.	Store the core in poly bags until primed. Then deliver the core to the QC station for approval.

INTERNAL INSULATION SYSTEM PROCESS PLAN

DATE (REV.) 5-9-72

S/A IDENT. ______ STEP 10 SHEET 1 of 1

SUBSTEP	OPERATION
	This step is for making the facesheet for A and B panels. Tools and special equipment required for this step are scissors and a humidity room. Materials required for this step are double-etched, 2-mil-thick Teflon film, 1-mil-thick Kapton film, cheesecloth, DC-1200 primer, and core assembly storage boxes.
a.	Cut the facesheet material (Kapton or Teflon) with scissors to the dimensions specified on the drawing.
b.	Place the facesheet on a clean sheet of polyethylene. Wipe the facesheet on both sides with toluene and allow to dry. Wipe the DC-1200 primer on both sides of the facesheet in two directions. Visually check for complete coverage as the primer is being applied.
c.	Allow the primer to air-dry for a minimum of 30 minutes at ambient conditions.
d.	Place the facesheet in the humidity room (minimum 40% RH) on a drying rack for a minimum of 3 hr.
e.	Visually inspect the sheet to verify that all areas of the sheet were covered.

INTERNAL INSULATION SYSTEM PROCESS PLAN

DATE (REV.) 5-9-72

S/A IDENT. ________ STEP 15 SHEET 1 of 1

SUBSTEP	OPERATION
	This step is for applying the DC-1200 primer to the core assembly. Tools and special equipment required for this step are the expansion tool (TX16), spray gun, storage box, and humidity room. Material required for this step is the DC-1200 primer. The core assembly (Step 5) is also required for this step.
a.	Remove a core assembly from the humidity room. Clean the core assembly by sloshing it in a tray filled with trichlorethylene. Shake off the excess liquid and assemble the panel immediately on the expansion tool (TX16). Allow the solvent to completely evaporate before proceeding to the next step.
b.	Spray the DC-1200 primer on the top surface of the core in four directions.
c.	Visually inspect the core to verify that all areas on the top surface are covered.
d.	Discard any unused primer and clean the spray gun with naphtha if no further priming is to be done within 1 hr. Store the gun with a small amount of naphtha in the can.
e.	Allow the core to air dry for 30 minutes at ambient conditions.
f.	Place the core in the humidity room and allow the primer to cure for a minimum of 6 hr.
g.	Store the core in a storage box in the humidity room or proceed to Step 25.

INTERNAL INSULATION SYSTEM PROCESS PLAN

DATE(REV.) 5-9-72

S/A IDENT. STEP 20 SHEET 1 of 1

SUBSTEP	OPERATION
	This step is for mixing the RTV-560 adhesive for bonding the core and facesheet. Equipment needed for this step are a paper mixing cup and spatula. Materials needed for this step are RTV-560 adhesive and standard catalyst (dibutyl tin dilaurate or Thermalite 12).
a.	Weigh the required quantity of RTV-560 in a paper mixing container.
b.	Add 0.25 percent by weight of standard catalyst to the RTV-560.
c.	Mix thoroughly by hand with a spatula for a minimum 5 minutes. (Note: This mixing time is appropriate for quantities of from 1 to 3 lb adhesive.) The adhesive should be used immediately.

DATE (REV.) 5-9-72

S/A IDENT. ______ STEP 25 SHEET 1 of 1

SUBSTEP	OPERATION
	This step is for bonding the facesheet to the core assembly of the A or B panels. Tools and special equipment needed for this step are the assembly tool (TX06). vacuum box lid (TX14), notched trowel, transfer sheet (TX15), vacuum pump system, and humidity room. Materials needed for this step are the mixed RTV-560 adhesive (Step 20) and a core assembly storage box. Completed subassemblies needed for this step are a core assembly (Step 5) and facesheet (Step 10). This entire step is performed in the humidity room.
a.	Remove [illegible] core assembly from the storag[illegible] n the humidity room and expand it on the assembly tool (TX06). [illegible] o verify that al[illegible] cells are properly fitted on the rake.
b.	Carefully place the transfer sheet (TX15) on the surface of the vacuum box lid (TX14).
c.	Spread a uniform thickness of 1/16 in. of RTV-560 on the transfer sheet with a notched trowel.
d.	Invert the assembly tool. Attach the assembly tool to the lid of the vacuum box.
e.	Open the vacuum valve to the vacuum box and evacuate the box to a vacuum of approximately 0.11 psi (3 in. of water column). This forces the adhesive up against the core. After 3 minutes. break the vacuum box back to ambient pressure.
f.	Remove the lid of the vacuum box and the transfer sheet from the base. Do not return the base to the upright orientation until substep i. Allow the core to drip for 10 minutes.
g.	Place a facesheet (from Step 10) on the vacuum box lid and reassemble the vacuum box.
h.	Evacuate the vacuum box to 0.50 psi (14 in. of water column) and maintain this vacuum for a minimum of 12 hr.
i.	Break the system back to ambient pressure and remove the lid of the vacuum box.
j.	Remove the panel from the assembly tool by loosening the edges of the facesheet and then lifting the panel with air pressure.
k.	Visually inspect the facesheet core bond for bonding integrity. If any part of the bondline is defective or questionable, the discrepancy shall be noted on a tag attached to the panel. The panel will be reviewed by the program manager for final disposition.
ℓ.	Place the panel in a storage box in the humidity room.

DATE (REV.) 5-9-72

S/A IDENT. ______ STEP 30 SHEET 1 of 1

SUBSTEP	OPERATION
	This step is for dimpling the facesheet of the A or B panels. Tools and special equipment required for this step are the dimpling heater,* thermocouple readout device, variable power transformer, vacuum pump system, and dimple depth tool (T001). Material needed for this step is a core assembly storage box. The completed subassembly needed for this step is a completed core and facesheet panel for Step 25. The panels should have been cured for 48 hr in the humidity room before dimpling.
a.	Remove a panel from the humidity room and install it in the dimpling/vacuum box (TX10) under the dimpling heater frame.
b.	Turn on both heaters to full power. Attach the vacuum lines and vacuum metering gage. Evacuate the vacuum box to between 0.32 and 0.36 psi (9 to 10 in. of water column).
c.	Measure the temperature of the dimpling heater with the thermocouple readout device held 2 in. in front of the heater. When the temperature reaches the 590-620°F range, swing the heater back and forth over the facesheet until the specified minimum dimple depth† is achieved. This requires approximately 1 minute.
d.	After the assembly has cooled to room temperature, break the system back to ambient pressure and remove the lid of the vacuum box.
e.	Inspect dimpling with the dimple depth tool (T001) to verify that each dimple exceeds a minimum depth of 1/8 in. If any dimples are less than the required depth, repeat substeps c and d.
f	Remove panel from vacuum box and store in storage box or proceed to Step 35.
	*A hand-held heater may be used for panels with unusual curvature.
	†The minimum dimple depth will vary, depending on cell size.

INTERNAL INSULATION SYSTEM PROCESS PLAN

DATE (REV.) 5-9-72

S/A IDENT. ________ STEP 35 SHEET 1 of 1

SUBSTEP	OPERATION
	This step is for perforating the facesheet of the A or B panels. Tools and special equipment required for this step are a soldering iron equipped with an 0.035-in. needle and a variable power supply. The completed subassembly required for this step is a dimpled panel from Step 30.
a.	Turn on the soldering iron to the preset power and allow the needle to heat. If the panel is stored, remove it from the storage box and install it in the vacuum box (TX10).
b.	Perforate the facesheet of each cell near its center. Before each erforating process, the operator shall practice perforating on a practice panel until consistently good results are achieved.
c.	Visually inspect the facesheet to verify that each cell has been perforated and that the hole is not enlarged. Repeat Substep b for any unperforated cells. If the perforation in any cell appears to be enlarged, note the cell(s) on a tag attached to the panel. The panel will be reviewed by the program manager for final disposition.
d.	Remove the panel from the vacuum box and either store it in the storage box or proceed to Step 40.

INTERNAL INSULATION SYSTEM PROCESS PLAN

DATE (REV.) 5-9-72

S/A IDENT. STEP 40 SHEET 1 of 1

SUBSTEP	OPERATION
	This step is for trimming the facesheet of the A or B panels. Tools and special equipment required for this step are a No. 11 X-Acto knife and a humidity room. The completed subassembly required for this step is a perforated panel from Step 35.
a.	If the panel is stored, remove it from the storage box and install it in the vacuum box (TX10).
b.	Using a No. 11 X-Acto knife, trim the excess facesheet material close to the perimeter of the core, removing approximately half of the outside bondline.
c.	Visually inspect the core assembly to verify that the facesheet was trimmed properly. Any discrepancies shall be noted in a tag attached to the panel. The panel will be reviewed by the program manager for final disposition.
d.	Remove the panel from the vacuum box and either store it in a storage box or proceed with Step 45.

INTERNAL INSULATION SYSTEM PROCESS PLAN

DATE (REV) 5-9-72

S/A IDENT. ______ STEP 45 SHEET 1 of 1

SUBSTEP	OPERATION
	This step is for making and installing the fiberglass filler plugs for the A or B panels. Tools and special equipment needed for this step are the filler plug die sets (TY17), punch press contour form (TX12), spray gun, and filler plug insertion tool (TY18). Materials needed for this step are rolls of 1/2-in. thick fiberglass, DC-1200 primer, and a core assembly storage bag. The completed subassembly required for this step is a trimmed panel (from Step 40).
a.	Place the halves of the filler plug die set (TY17) in the punch press and punch the required number of filler plugs according to the panel drawing.
b.	Clean the open side of the core by sloshing it in a tray filled with trichloroethylene. Shake off the excess liquid and allow the core to dry.
c.	Position a panel on the contour form (TX12) with the facesheet against the core.
d.	Install the filler plugs using the insertion tool (TY18). Two 1/2-in.-thick plugs are inserted into each cell.
e.	Inspect each cell to verify that the outermost surface of the filler plug in each cell is depressed between 1/32 and 1/16 in. below the outside edge of the core. Adjust any plugs as required.
g.	Using the spray gun, apply the DC-1200 primer to the exposed area of the core and to the outside perimeter of the panel.
h.	Allow the primer to air dry for a minimum of 30 minutes at ambient conditions.
i.	Place the panel in the humidity room (minimum 40% RH) and allow the primer to cure for a minimum of 3 hr.
j.	After the primer has cured, visually inspect the assembly to verify that each filler plug is bonded in place between 1/32 and 1/16 in. below the edge of the core. Adjust any plugs that are depressed too much or too little and brush on primer to cement the plugs in place.
k.	Place the completed assembly in a storage bog or box. The completed panel is now ready for installation on the test tank.

APPENDIX C

PANEL INSPECTION RECORD
AND QUALITY CONTROL CRITERIA

PANEL INSPECTION RECORD

PANEL ID________________

A. CORE FABRICATION

1. Verify ID marked on Core ________
2. Node Bonds in Tolerance ________
3. Short Node Bonds ________
4. Skips in Node Bonds ________
5. Bond Line Thickness ________
6. Ribbons Tears ________
7. Ribbon Pattern ________
8. Trim Outline in Tolerance ________
9. Trim Finish ________
10. End Trim in Tolerance ________
11. Ribbon Alignment ________

OK FOR STEPS 10 & 15

By________________Date________

B. PRIMING

1. Core Priming ________
2. Facesheet Priming ________
3. Facesheet Identified ________

OK FOR STEP 25

By________________Date________

C. FACE SHEET ASSEMBLY

1. Bond/Primer Integrity ________
2. Bond Line Width ________
3. Bond Line Skips ________
4. Voids at Core Node ________
5. Cell Distortion ________
6. Wrinkles ________
7. Ribbon Tears ________

OK FOR STEP 30

By________________Date________

D. DIMPLING

1. Verify Dimpling Parameters ________
2. Dimple Depth ________
3. Dimple Into Corners ________
4. Holes in Facesheet ________

OK FOR STEPS 35 & 40

By________________Date________

E. PERFORATING & TRIMMING

1. Hole Size ________
2. Hole Uniformity ________
   a. Hole Shape ________
   b. Tearing ________
   c. All Cells Perforated ________
3. Trim ________
   a. Damage to Perimeter Bond ________
   b. Overtrim ________
   c. Undertrim ________

OK FOR STEP 45

By________________Date________

F. FINAL INSPECTION

1. Filler Plug Installation ________
2. Back Side Priming ________
3. Overall Appearance ________

PANEL READY FOR INSTALLATION

By________________Date________

QC CRITERIA FOR INTERNAL INSULATION

A. CORE FABRICATION

1. The Panel ID number on the core must be clearly legible, and be marked on the side of core at one corner.
2. The width of node bond lines must be in accordance with the fabrication drawing, to within ±1/16 in.
3. Node bonds must be complete to within 0.005 in. of the ribbon edge on both sides.
4. Node bonds must be free of voids that traverse more than 1/4 the width of the bond line.
5. The thickness of the bond line must be measured at a minimum of four places on both sides of core assembly. Record the minimum and maximum bond thickness (obtained by subtracting the thickness of two ribbons).
6. Ribbon tears - Core ribbons must be free of tears on both sides.
7. The ribbon pattern (order of assembly) must be in accordance with the fabrication drawing.
8. The trim outline (for contoured panels) must be in accordance with the master drawing within ±1/32 in. at all places.
9. Trimmed core surfaces must be smooth and free of discontinuities in excess of 0.005 in.
10. End node bonds must be trimmed in accordance with the panel drawing to within ±1/32 in.
11. Edges of adjacent core ribbons must not mismatch in alignment by more than 0.005 in. on both sides.

B. PRIMING

1. Core Priming

   a. Verify that the proper cure cycle has been completed.
   b. Verify by visual inspection that primer has been applied to all of the core surface to within 1/4 in. of the edge of the facesheet.

2. Facesheet Priming

   a. Verify by visual inspection that primer has been applied to both sides of the Teflon facesheet.
   b. Verify that the proper cure cycle has been completed.

3. ID - If not present already, apply a panel identification number to the corner of the facesheet using a black felt-tip marker.

C. FACESHEET ASSEMBLY

1. Verify the integrity of the primer and adhesive bond by failing a short portion of the bond (approximately 1/4 in.) at each of the untrimmed corner tabs.
2. Inspect the width of the bond lines at the facesheet. If the bond width is less than 0.020 in., leak-check the affected cells to ensure that the bond is complete. If the width exceeds 0.200 in., inspect further to determine whether a satisfactory dimple will be obtained.
3. Discontinuities or skips in bond lines on either side of the core ribbon are unacceptable.
4. Inspect the core node bonds for voids in the bond lines.
5. Perimeter cells shall not be distorted beyond limits as indicated by gages. Internal cells shall not be distorted so as to cause straight cell walls.
6. Wrinkling of the facesheet shall not cause leakage between cells.
7. The core ribbons shall not be torn.

D. DIMPLING

1. Verify that the dimpling temperature and pressure parameters recorded on the process record sheet are in accordance with current procedure and properly signed off by the operator.

2. Measure the depth of the dimple for at least 20 cells, including cells adjacent to all edges. 90% of the cells so measured must have a minimum dimple depth of 5/32 in. No cell shall have a dimple depth less than 1/8 in. (Note - These criteria are for cells approximately 2 in. high by 1.75 in. wide and 1 in. in panel thickness.)

3. Verify by visual inspection that the dimple extends well into the corners of each cell.

4. Inspect the entire facesheet to verify that there are no holes.

E. PERFORATING AND TRIMMING

1. Measure the size of at least 20 holes in the facesheet. Each hole must be between 0.025 and 0.045 in. in diameter.

2. Visually inspect the entire facesheet to verify--

   a. that the holes are round, to the extent that the maximum and minimum dimensions are within the tolerances specified in (1) above.
   b. that the holes and facesheet are free of tears.
   c. that all cells have been perforated.

3. Inspect the entire perimeter to verify that--

   a. the facesheet-to-core bond is intact.
   b. adhesive is not completely removed, at any point, from the outside of the core ribbons.
   c. the maximum amount of facesheet and adhesive remaining beyond the core ribbon does not exceed 1/32 in.

F. FINAL INSPECTION

1. Inspect the panel on both sides to verify that all filler plugs are installed properly, and that--

   a. no gaps or cocked plugs are visible.
   b. all filler plugs are depressed between 1/32 and 1/8 in. below the back of the core, and that no filler material extends across any cell wall.

2. Verify that the primer has been applied uniformly to the back side of the panel.

3. Perform an overall inspection to detect any defects that may be visible and to verify that the panel has an acceptable appearance.